

Cover page for Supplementary Information

Manuscript Title:

Formation of stable enols from 1,4-dithio-1,3-dienes and acid chlorides by a de-aromatization/Michael addition/re-aromatization domino process

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Contents:

- 1. Crystallographic data and lists of bond lengths and angles for 3a.**
- 2. Copies of ^1H NMR and ^{13}C NMR spectra for all new compounds.**

1. Crystallographic data and lists of bond lengths and angles for 3a.

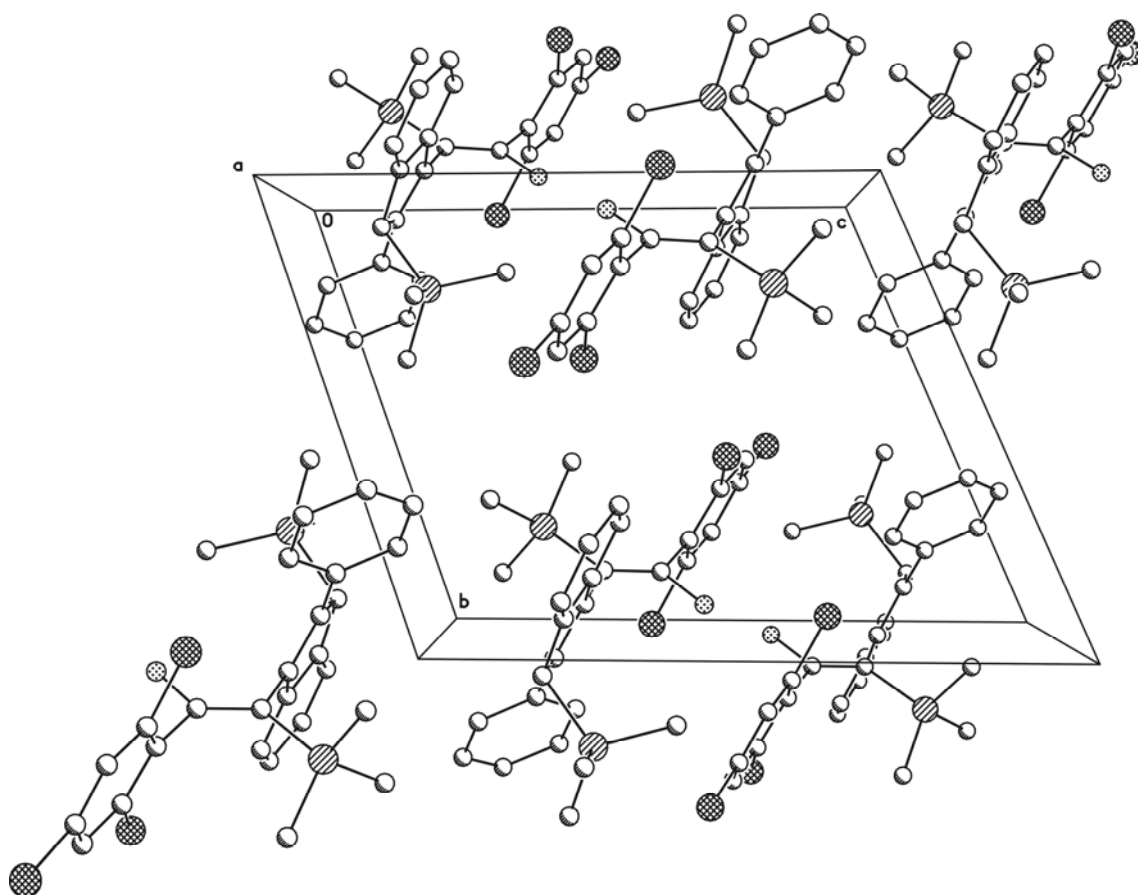
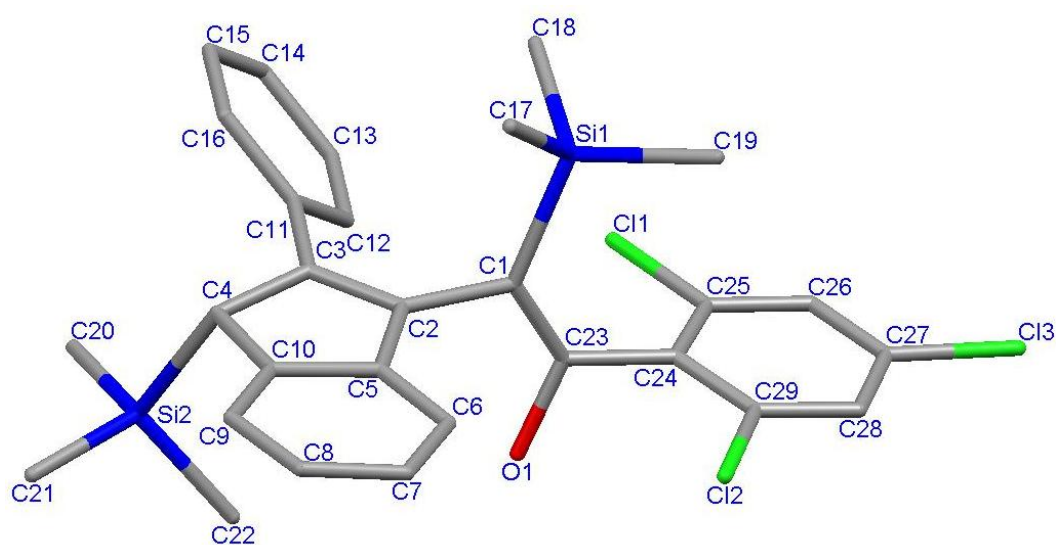


Table 1. Crystal data and structure refinement for **3a**.

Identification code	a
Empirical formula	C ₂₉ H ₃₁ Cl ₃ O Si ₂
Formula weight	558.07
Temperature	293(2) K
Measurement device	Rigaku RAXIS RAPID IP
Measurement method	\W
Wavelength	0.71073 Å
Program for data collection	Rapid-AUTO (Rigaku, 2000)
Program for cell refinement	Rapid-AUTO
Program for data reduction	CrystalStructure (Rigaku/MS, 2000)
Program for structure solution	SHELXS-97, (Sheldrick, 1997)
Program for structure refinement	SHELXL-97, (Sheldrick, 1997)
Program for molecular graphics	Siemens SHELXTL V4.2, (Sheldrick, 1990)
Program for publication material	SHELXL-97, (Sheldrick, 1997)
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 11.237(2) Å alpha = 68.38(3) deg. b = 11.608(2) Å beta = 84.74(3) deg. c = 13.253(3) Å gamma = 66.43(3) deg.
Volume	1470.0(5) Å ³
Refls. No. for cell measurement	10269
Theta range for cell measurement	2.37 to 25.03 deg.
Z, Calculated density	2, 1.261 Mg/m ³
Absorption coefficient	0.414 mm ⁻¹
F(000)	584
Crystal shape / Crystal colour	block / colourless
Crystal size	0.50 x 0.45 x 0.15 mm
Theta range for data collection	2.37 to 25.03 deg.
Limiting indices	-13<=h<=12, -13<=k<=13, -15<=l<=14
Reflections collected / unique	10269 / 5090 [R(int) = 0.0580]
Reflections with I>2sigma(I)	3220
Completeness to theta = 25.03	97.8 %
Decay correction (%)	0
Absorption correction	Empirical
Max. and min. transmission	0.940 and 0.813
Method for primary solution	direct
Method for secondary solution	difmap
Hydrogen addition / treatment	geom or difmap / mixed
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5090 / 0 / 321
Goodness-of-fit on F ²	1.002
Final R indices [I>2sigma(I)]	R1 = 0.0491, wR2 = 0.1193
R indices (all data)	R1 = 0.0790, wR2 = 0.1280
Extinction coefficient	0.025(2)
Largest diff. peak and hole	0.240 and -0.355 e.Å ⁻³
Max. and mean shift/sigma	0.000 and 0.000

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3a**.
 $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
Cl (1)	−323 (1)	10128 (1)	3429 (1)	65 (1)
Cl (2)	3980 (1)	6099 (1)	5870 (1)	68 (1)
Cl (3)	−702 (1)	5932 (1)	6732 (1)	84 (1)
Si (1)	2760 (1)	7725 (1)	2276 (1)	52 (1)
Si (2)	4869 (1)	12269 (1)	1870 (1)	56 (1)
O (1)	2918 (2)	9424 (2)	4526 (2)	59 (1)
C (1)	3069 (3)	8675 (2)	3041 (2)	40 (1)
C (2)	3989 (3)	9350 (3)	2541 (2)	42 (1)
C (3)	3761 (3)	10536 (3)	1700 (2)	43 (1)
C (4)	4987 (3)	10800 (3)	1468 (2)	47 (1)
C (5)	5388 (3)	8697 (3)	2879 (2)	43 (1)
C (6)	6105 (3)	7476 (3)	3695 (2)	54 (1)
C (7)	7432 (3)	7071 (3)	3806 (3)	65 (1)
C (8)	8043 (3)	7842 (3)	3120 (3)	66 (1)
C (9)	7343 (3)	9074 (3)	2315 (3)	59 (1)
C (10)	6005 (3)	9510 (3)	2205 (2)	45 (1)
C (11)	2505 (3)	11498 (3)	1087 (2)	47 (1)
C (12)	1373 (3)	11880 (3)	1616 (3)	60 (1)
C (13)	205 (4)	12787 (3)	1047 (3)	79 (1)
C (14)	155 (5)	13322 (4)	−78 (4)	94 (1)
C (15)	1261 (5)	12973 (4)	−626 (3)	89 (1)
C (16)	2438 (4)	12054 (3)	−48 (3)	68 (1)
C (17)	4292 (4)	7065 (4)	1608 (3)	75 (1)
C (18)	1408 (4)	8846 (4)	1203 (3)	104 (2)
C (19)	2402 (4)	6251 (3)	3175 (3)	77 (1)
C (20)	3575 (4)	13866 (3)	999 (3)	82 (1)
C (21)	6444 (3)	12479 (4)	1693 (3)	81 (1)
C (22)	4463 (4)	11860 (4)	3324 (3)	81 (1)
C (23)	2634 (3)	8724 (3)	3998 (2)	43 (1)
C (24)	1781 (3)	8067 (3)	4676 (2)	43 (1)
C (25)	436 (3)	8606 (3)	4491 (2)	47 (1)
C (26)	−336 (3)	7976 (3)	5123 (2)	52 (1)
C (27)	242 (3)	6777 (3)	5969 (2)	55 (1)
C (28)	1570 (3)	6213 (3)	6214 (2)	53 (1)
C (29)	2312 (3)	6861 (3)	5571 (2)	49 (1)

Table 3. Bond lengths [Å] and angles [deg] for **3a**.

Cl (1)–C (25)	1.735 (3)
Cl (2)–C (29)	1.733 (3)
Cl (3)–C (27)	1.736 (3)
Si (1)–C (19)	1.863 (3)
Si (1)–C (18)	1.864 (4)
Si (1)–C (17)	1.871 (4)
Si (1)–C (1)	1.887 (3)
Si (2)–C (21)	1.863 (3)
Si (2)–C (20)	1.860 (3)
Si (2)–C (22)	1.866 (4)
Si (2)–C (4)	1.919 (3)
O (1)–C (23)	1.384 (3)
O (1)–H01	0.82 (4)
C (1)–C (23)	1.330 (4)
C (1)–C (2)	1.503 (4)
C (2)–C (3)	1.359 (4)
C (2)–C (5)	1.474 (4)
C (3)–C (11)	1.476 (4)
C (3)–C (4)	1.508 (4)
C (4)–C (10)	1.504 (4)
C (4)–H (4)	0.9800
C (5)–C (6)	1.395 (4)
C (5)–C (10)	1.399 (4)
C (6)–C (7)	1.376 (4)
C (6)–H (6)	0.9300
C (7)–C (8)	1.370 (5)
C (7)–H (7)	0.9300
C (8)–C (9)	1.390 (4)
C (8)–H (8)	0.9300
C (9)–C (10)	1.384 (4)
C (9)–H (9)	0.9300
C (11)–C (12)	1.385 (4)
C (11)–C (16)	1.398 (4)
C (12)–C (13)	1.376 (4)
C (12)–H (12)	0.9300
C (13)–C (14)	1.384 (5)
C (13)–H (13)	0.9300
C (14)–C (15)	1.373 (6)
C (14)–H (14)	0.9300
C (15)–C (16)	1.389 (5)
C (15)–H (15)	0.9300
C (16)–H (16)	0.9300
C (17)–H (17A)	0.9600
C (17)–H (17B)	0.9600
C (17)–H (17C)	0.9600
C (18)–H (18A)	0.9600
C (18)–H (18B)	0.9600
C (18)–H (18C)	0.9600
C (19)–H (19A)	0.9600

C (19)–H (19B)	0.9600
C (19)–H (19C)	0.9600
C (20)–H (20A)	0.9600
C (20)–H (20B)	0.9600
C (20)–H (20C)	0.9600
C (21)–H (21A)	0.9600
C (21)–H (21B)	0.9600
C (21)–H (21C)	0.9600
C (22)–H (22A)	0.9600
C (22)–H (22B)	0.9600
C (22)–H (22C)	0.9600
C (23)–C (24)	1.494 (4)
C (24)–C (25)	1.391 (4)
C (24)–C (29)	1.400 (4)
C (25)–C (26)	1.383 (4)
C (26)–C (27)	1.371 (4)
C (26)–H (26)	0.9300
C (27)–C (28)	1.383 (4)
C (28)–C (29)	1.374 (4)
C (28)–H (28)	0.9300

C (19)–Si (1)–C (18)	108.92 (19)
C (19)–Si (1)–C (17)	107.50 (16)
C (18)–Si (1)–C (17)	108.66 (19)
C (19)–Si (1)–C (1)	113.25 (14)
C (18)–Si (1)–C (1)	111.73 (15)
C (17)–Si (1)–C (1)	106.58 (15)
C (21)–Si (2)–C (20)	108.49 (17)
C (21)–Si (2)–C (22)	110.51 (18)
C (20)–Si (2)–C (22)	110.13 (19)
C (21)–Si (2)–C (4)	110.27 (15)
C (20)–Si (2)–C (4)	110.17 (15)
C (22)–Si (2)–C (4)	107.28 (14)
C (23)–O (1)–H01	112 (3)
C (23)–C (1)–C (2)	117.7 (2)
C (23)–C (1)–Si (1)	126.8 (2)
C (2)–C (1)–Si (1)	115.3 (2)
C (3)–C (2)–C (5)	108.5 (2)
C (3)–C (2)–C (1)	129.3 (2)
C (5)–C (2)–C (1)	122.0 (2)
C (2)–C (3)–C (11)	126.9 (3)
C (2)–C (3)–C (4)	110.7 (2)
C (11)–C (3)–C (4)	122.4 (2)
C (10)–C (4)–C (3)	102.8 (2)
C (10)–C (4)–Si (2)	109.47 (19)
C (3)–C (4)–Si (2)	111.0 (2)
C (10)–C (4)–H (4)	111.1
C (3)–C (4)–H (4)	111.1
Si (2)–C (4)–H (4)	111.1
C (6)–C (5)–C (10)	120.5 (3)
C (6)–C (5)–C (2)	130.8 (3)

C(10)–C(5)–C(2)	108.7(2)
C(7)–C(6)–C(5)	118.8(3)
C(7)–C(6)–H(6)	120.6
C(5)–C(6)–H(6)	120.6
C(8)–C(7)–C(6)	120.8(3)
C(8)–C(7)–H(7)	119.6
C(6)–C(7)–H(7)	119.6
C(7)–C(8)–C(9)	121.3(3)
C(7)–C(8)–H(8)	119.4
C(9)–C(8)–H(8)	119.4
C(10)–C(9)–C(8)	118.8(3)
C(10)–C(9)–H(9)	120.6
C(8)–C(9)–H(9)	120.6
C(9)–C(10)–C(5)	119.9(3)
C(9)–C(10)–C(4)	131.3(3)
C(5)–C(10)–C(4)	108.8(2)
C(12)–C(11)–C(16)	118.5(3)
C(12)–C(11)–C(3)	121.3(3)
C(16)–C(11)–C(3)	120.2(3)
C(13)–C(12)–C(11)	121.4(3)
C(13)–C(12)–H(12)	119.3
C(11)–C(12)–H(12)	119.3
C(12)–C(13)–C(14)	119.4(4)
C(12)–C(13)–H(13)	120.3
C(14)–C(13)–H(13)	120.3
C(15)–C(14)–C(13)	120.7(4)
C(15)–C(14)–H(14)	119.7
C(13)–C(14)–H(14)	119.7
C(14)–C(15)–C(16)	119.8(4)
C(14)–C(15)–H(15)	120.1
C(16)–C(15)–H(15)	120.1
C(15)–C(16)–C(11)	120.3(4)
C(15)–C(16)–H(16)	119.8
C(11)–C(16)–H(16)	119.8
Si(1)–C(17)–H(17A)	109.5
Si(1)–C(17)–H(17B)	109.5
H(17A)–C(17)–H(17B)	109.5
Si(1)–C(17)–H(17C)	109.5
H(17A)–C(17)–H(17C)	109.5
H(17B)–C(17)–H(17C)	109.5
Si(1)–C(18)–H(18A)	109.5
Si(1)–C(18)–H(18B)	109.5
H(18A)–C(18)–H(18B)	109.5
Si(1)–C(18)–H(18C)	109.5
H(18A)–C(18)–H(18C)	109.5
H(18B)–C(18)–H(18C)	109.5
Si(1)–C(19)–H(19A)	109.5
Si(1)–C(19)–H(19B)	109.5
H(19A)–C(19)–H(19B)	109.5
Si(1)–C(19)–H(19C)	109.5
H(19A)–C(19)–H(19C)	109.5

H(19B)–C(19)–H(19C)	109.5
Si(2)–C(20)–H(20A)	109.5
Si(2)–C(20)–H(20B)	109.5
H(20A)–C(20)–H(20B)	109.5
Si(2)–C(20)–H(20C)	109.5
H(20A)–C(20)–H(20C)	109.5
H(20B)–C(20)–H(20C)	109.5
Si(2)–C(21)–H(21A)	109.5
Si(2)–C(21)–H(21B)	109.5
H(21A)–C(21)–H(21B)	109.5
Si(2)–C(21)–H(21C)	109.5
H(21A)–C(21)–H(21C)	109.5
H(21B)–C(21)–H(21C)	109.5
Si(2)–C(22)–H(22A)	109.5
Si(2)–C(22)–H(22B)	109.5
H(22A)–C(22)–H(22B)	109.5
Si(2)–C(22)–H(22C)	109.5
H(22A)–C(22)–H(22C)	109.5
H(22B)–C(22)–H(22C)	109.5
C(1)–C(23)–O(1)	123.9(3)
C(1)–C(23)–C(24)	127.3(3)
O(1)–C(23)–C(24)	108.8(2)
C(25)–C(24)–C(29)	115.9(3)
C(25)–C(24)–C(23)	123.5(2)
C(29)–C(24)–C(23)	120.6(2)
C(26)–C(25)–C(24)	122.6(3)
C(26)–C(25)–Cl(1)	117.9(2)
C(24)–C(25)–Cl(1)	119.5(2)
C(27)–C(26)–C(25)	118.9(3)
C(27)–C(26)–H(26)	120.6
C(25)–C(26)–H(26)	120.6
C(26)–C(27)–C(28)	121.2(3)
C(26)–C(27)–Cl(3)	119.5(3)
C(28)–C(27)–Cl(3)	119.3(2)
C(29)–C(28)–C(27)	118.6(3)
C(29)–C(28)–H(28)	120.7
C(27)–C(28)–H(28)	120.7
C(28)–C(29)–C(24)	122.8(3)
C(28)–C(29)–Cl(2)	117.4(2)
C(24)–C(29)–Cl(2)	119.7(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3a**.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
C1(1)	49(1)	62(1)	74(1)	-9(1)	-11(1)	-21(1)
C1(2)	50(1)	64(1)	75(1)	-6(1)	-10(1)	-21(1)
C1(3)	77(1)	90(1)	91(1)	-20(1)	22(1)	-56(1)
Si(1)	57(1)	54(1)	58(1)	-22(1)	-2(1)	-31(1)
Si(2)	55(1)	51(1)	69(1)	-19(1)	0(1)	-31(1)
O(1)	67(2)	74(2)	65(1)	-36(1)	11(1)	-46(1)
C(1)	36(2)	37(1)	47(2)	-11(1)	-4(1)	-18(1)
C(2)	44(2)	42(2)	46(2)	-13(1)	-2(1)	-23(1)
C(3)	44(2)	47(2)	45(2)	-16(1)	-2(1)	-23(1)
C(4)	46(2)	54(2)	45(2)	-14(1)	6(1)	-28(1)
C(5)	43(2)	39(2)	52(2)	-20(1)	-2(1)	-17(1)
C(6)	53(2)	45(2)	62(2)	-14(2)	-6(2)	-21(2)
C(7)	55(2)	55(2)	80(2)	-22(2)	-17(2)	-17(2)
C(8)	41(2)	69(2)	92(3)	-39(2)	-7(2)	-14(2)
C(9)	48(2)	68(2)	75(2)	-36(2)	12(2)	-31(2)
C(10)	42(2)	49(2)	53(2)	-21(1)	3(1)	-23(1)
C(11)	52(2)	39(2)	55(2)	-14(1)	-10(2)	-22(1)
C(12)	56(2)	51(2)	64(2)	-11(2)	-10(2)	-19(2)
C(13)	56(2)	65(2)	101(3)	-24(2)	-11(2)	-11(2)
C(14)	80(3)	71(3)	107(4)	-23(3)	-43(3)	-5(2)
C(15)	103(3)	74(3)	67(2)	-13(2)	-33(2)	-17(2)
C(16)	78(3)	64(2)	53(2)	-16(2)	-11(2)	-22(2)
C(17)	93(3)	88(3)	67(2)	-38(2)	25(2)	-55(2)
C(18)	111(4)	92(3)	114(3)	-43(3)	-53(3)	-28(3)
C(19)	88(3)	81(2)	101(3)	-54(2)	35(2)	-59(2)
C(20)	76(3)	52(2)	118(3)	-28(2)	-7(2)	-25(2)
C(21)	67(2)	77(2)	115(3)	-31(2)	1(2)	-46(2)
C(22)	103(3)	90(3)	79(3)	-42(2)	12(2)	-57(2)
C(23)	39(2)	42(2)	51(2)	-14(1)	-7(1)	-18(1)
C(24)	41(2)	44(2)	48(2)	-18(1)	4(1)	-21(1)
C(25)	44(2)	47(2)	53(2)	-19(1)	-1(1)	-20(1)
C(26)	40(2)	62(2)	62(2)	-27(2)	8(2)	-26(2)
C(27)	61(2)	59(2)	60(2)	-26(2)	16(2)	-36(2)
C(28)	55(2)	53(2)	53(2)	-16(2)	7(2)	-28(2)
C(29)	44(2)	53(2)	55(2)	-21(2)	1(1)	-22(2)

Table 5. Torsion angles [deg] for **3a**.

C(19)–Si(1)–C(1)–C(23)	–25.3(3)
C(18)–Si(1)–C(1)–C(23)	98.1(3)
C(17)–Si(1)–C(1)–C(23)	–143.3(3)
C(19)–Si(1)–C(1)–C(2)	149.6(2)
C(18)–Si(1)–C(1)–C(2)	–86.9(3)
C(17)–Si(1)–C(1)–C(2)	31.6(2)
C(23)–C(1)–C(2)–C(3)	–104.7(3)
Si(1)–C(1)–C(2)–C(3)	79.9(3)
C(23)–C(1)–C(2)–C(5)	80.6(3)
Si(1)–C(1)–C(2)–C(5)	–94.8(3)
C(5)–C(2)–C(3)–C(11)	178.0(3)
C(1)–C(2)–C(3)–C(11)	2.7(5)
C(5)–C(2)–C(3)–C(4)	–3.9(3)
C(1)–C(2)–C(3)–C(4)	–179.1(3)
C(2)–C(3)–C(4)–C(10)	6.9(3)
C(11)–C(3)–C(4)–C(10)	–174.8(2)
C(2)–C(3)–C(4)–Si(2)	–110.1(2)
C(11)–C(3)–C(4)–Si(2)	68.2(3)
C(21)–Si(2)–C(4)–C(10)	62.2(2)
C(20)–Si(2)–C(4)–C(10)	–178.1(2)
C(22)–Si(2)–C(4)–C(10)	–58.2(2)
C(21)–Si(2)–C(4)–C(3)	175.0(2)
C(20)–Si(2)–C(4)–C(3)	–65.3(2)
C(22)–Si(2)–C(4)–C(3)	54.6(2)
C(3)–C(2)–C(5)–C(6)	–179.8(3)
C(1)–C(2)–C(5)–C(6)	–4.1(5)
C(3)–C(2)–C(5)–C(10)	–1.1(3)
C(1)–C(2)–C(5)–C(10)	174.6(2)
C(10)–C(5)–C(6)–C(7)	–1.6(4)
C(2)–C(5)–C(6)–C(7)	177.0(3)
C(5)–C(6)–C(7)–C(8)	–0.9(5)
C(6)–C(7)–C(8)–C(9)	2.1(5)
C(7)–C(8)–C(9)–C(10)	–0.8(5)
C(8)–C(9)–C(10)–C(5)	–1.6(4)
C(8)–C(9)–C(10)–C(4)	176.4(3)
C(6)–C(5)–C(10)–C(9)	2.9(4)
C(2)–C(5)–C(10)–C(9)	–176.1(3)
C(6)–C(5)–C(10)–C(4)	–175.6(3)
C(2)–C(5)–C(10)–C(4)	5.5(3)
C(3)–C(4)–C(10)–C(9)	174.4(3)
Si(2)–C(4)–C(10)–C(9)	–67.6(4)
C(3)–C(4)–C(10)–C(5)	–7.4(3)
Si(2)–C(4)–C(10)–C(5)	110.6(2)
C(2)–C(3)–C(11)–C(12)	46.1(4)
C(4)–C(3)–C(11)–C(12)	–131.9(3)
C(2)–C(3)–C(11)–C(16)	–134.5(3)
C(4)–C(3)–C(11)–C(16)	47.5(4)
C(16)–C(11)–C(12)–C(13)	–0.2(5)

C (3)–C (11)–C (12)–C (13)	179.3 (3)
C (11)–C (12)–C (13)–C (14)	0.5 (5)
C (12)–C (13)–C (14)–C (15)	–1.1 (6)
C (13)–C (14)–C (15)–C (16)	1.3 (6)
C (14)–C (15)–C (16)–C (11)	–1.0 (6)
C (12)–C (11)–C (16)–C (15)	0.4 (5)
C (3)–C (11)–C (16)–C (15)	–179.1 (3)
C (2)–C (1)–C (23)–O (1)	3.9 (4)
Si (1)–C (1)–C (23)–O (1)	178.78 (19)
C (2)–C (1)–C (23)–C (24)	–176.3 (2)
Si (1)–C (1)–C (23)–C (24)	–1.4 (4)
C (1)–C (23)–C (24)–C (25)	–81.2 (4)
O (1)–C (23)–C (24)–C (25)	98.7 (3)
C (1)–C (23)–C (24)–C (29)	100.5 (3)
O (1)–C (23)–C (24)–C (29)	–79.6 (3)
C (29)–C (24)–C (25)–C (26)	–2.2 (4)
C (23)–C (24)–C (25)–C (26)	179.5 (3)
C (29)–C (24)–C (25)–Cl (1)	177.9 (2)
C (23)–C (24)–C (25)–Cl (1)	–0.5 (4)
C (24)–C (25)–C (26)–C (27)	0.3 (4)
Cl (1)–C (25)–C (26)–C (27)	–179.7 (2)
C (25)–C (26)–C (27)–C (28)	1.8 (5)
C (25)–C (26)–C (27)–Cl (3)	–177.0 (2)
C (26)–C (27)–C (28)–C (29)	–1.8 (5)
Cl (3)–C (27)–C (28)–C (29)	176.9 (2)
C (27)–C (28)–C (29)–C (24)	–0.1 (5)
C (27)–C (28)–C (29)–Cl (2)	–177.8 (2)
C (25)–C (24)–C (29)–C (28)	2.1 (4)
C (23)–C (24)–C (29)–C (28)	–179.5 (3)
C (25)–C (24)–C (29)–Cl (2)	179.7 (2)
C (23)–C (24)–C (29)–Cl (2)	–1.9 (4)

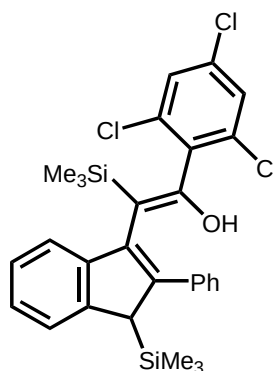
Symmetry transformations used to generate equivalent atoms:

2. Copies of ^1H NMR and ^{13}C NMR spectra for all new compounds

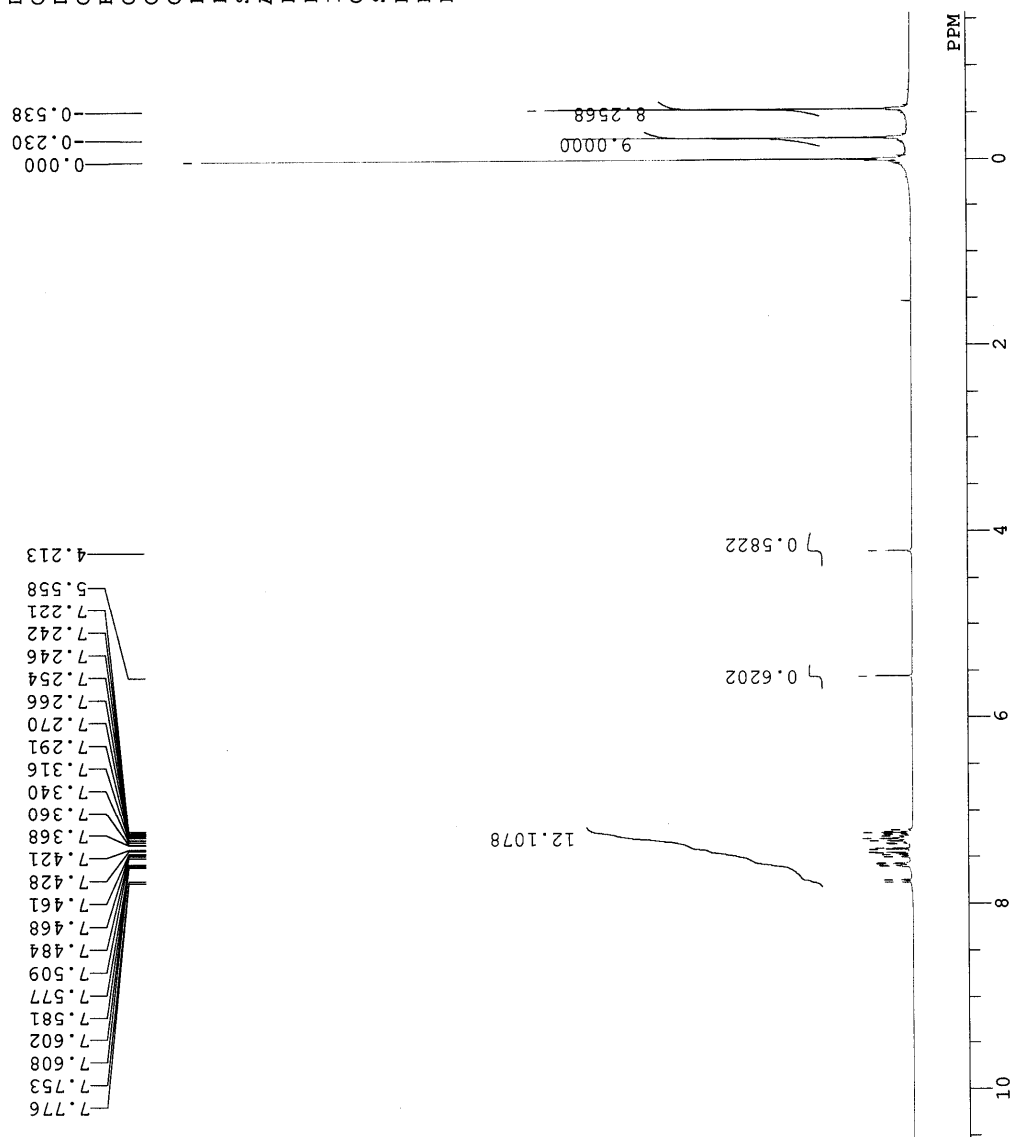
3a-HNMR-with TMS

D:\Huglaoshu\R499\H-TMS.als

Mon Jan 15 15:07:04 2007
 1H
 NON
 300.40 MHz
 130.00 KHz
 1150.0 Hz
 32768
 6013.2 Hz
 8
 5.449 sec
 1.551 sec
 5.8 us
 1H
 23.4 c
 CDCL3
 0.00 ppm
 0.09 Hz
 18
 RGAIN



3a



3a-HNMR-no-TMS

D:\Huqiaoshu\En-ol\H.als

DFILE

COMNT

DATIM

OBNUC

EXMOD

OBFRQ

OBSET

OBFIN

POINT

FREQU

SCANS

ACQTM

PD

PWL

IRNUC

CTEMP

SLVNT

EXREF

BF

RGAIN

Mon Jan 08 22:17:10 2007

1H

NON

300.40 MHz

130.00 KHz

1150.0 Hz

32768

6013.2 Hz

8

5.449 sec

1.551 sec

5.8 us

1H

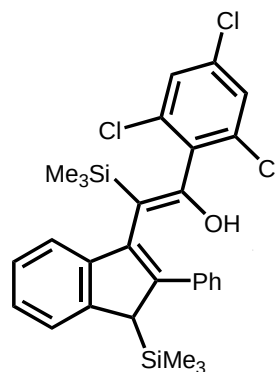
24.1 C

CDCL3

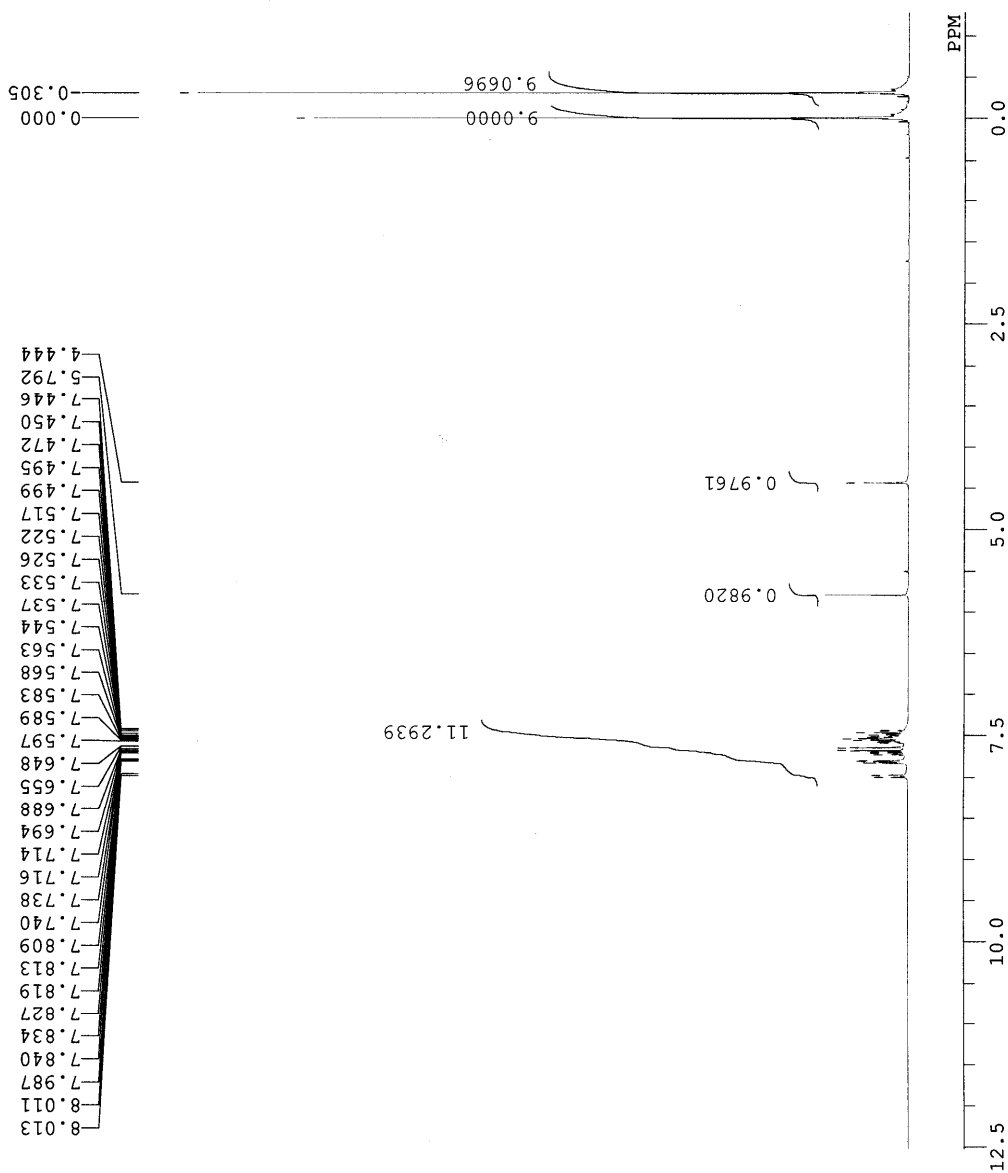
0.00 ppm

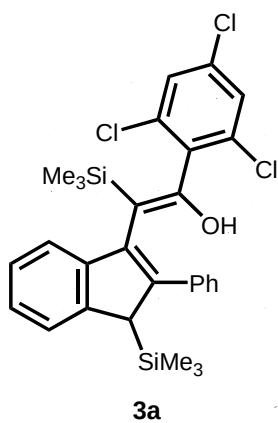
0.09 Hz

14



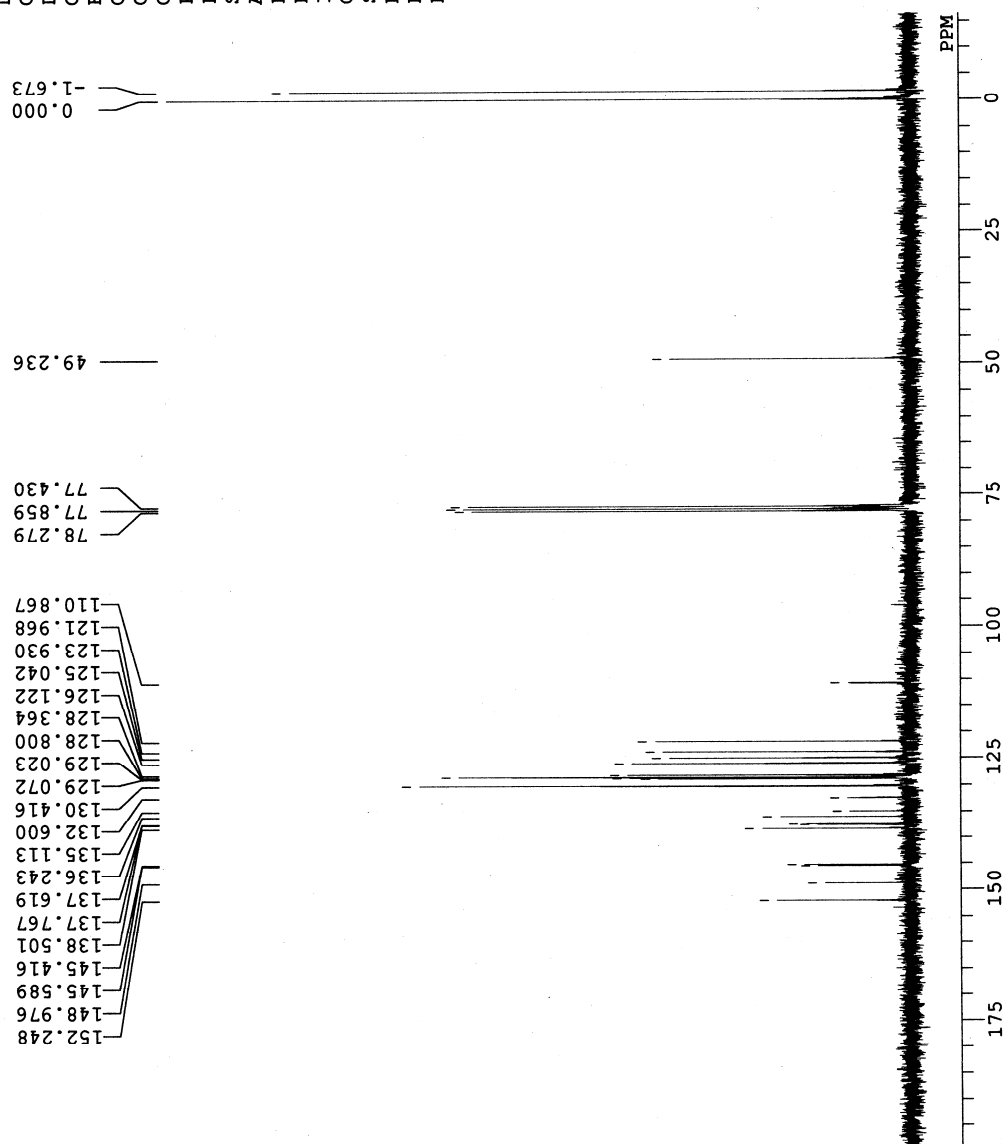
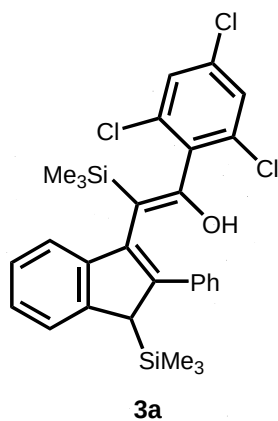
3a





3a-CNMR-no-TMS

D:\Hugiaoshu\R499\C.a.als
 COMNT Wed Nov 22 12:27:21 2006
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFRQ 124.00 KHz
 OBSET 1840.0 Hz
 OBFIN 32768
 POINT 20408.1 Hz
 FREQU 576
 SCANS 1.606 sec
 ACQTM 1.394 sec
 PD 5.0 us
 PW1 1H
 IRNUC 24.7 c
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.62 Hz
 BF 26
 RGAIN

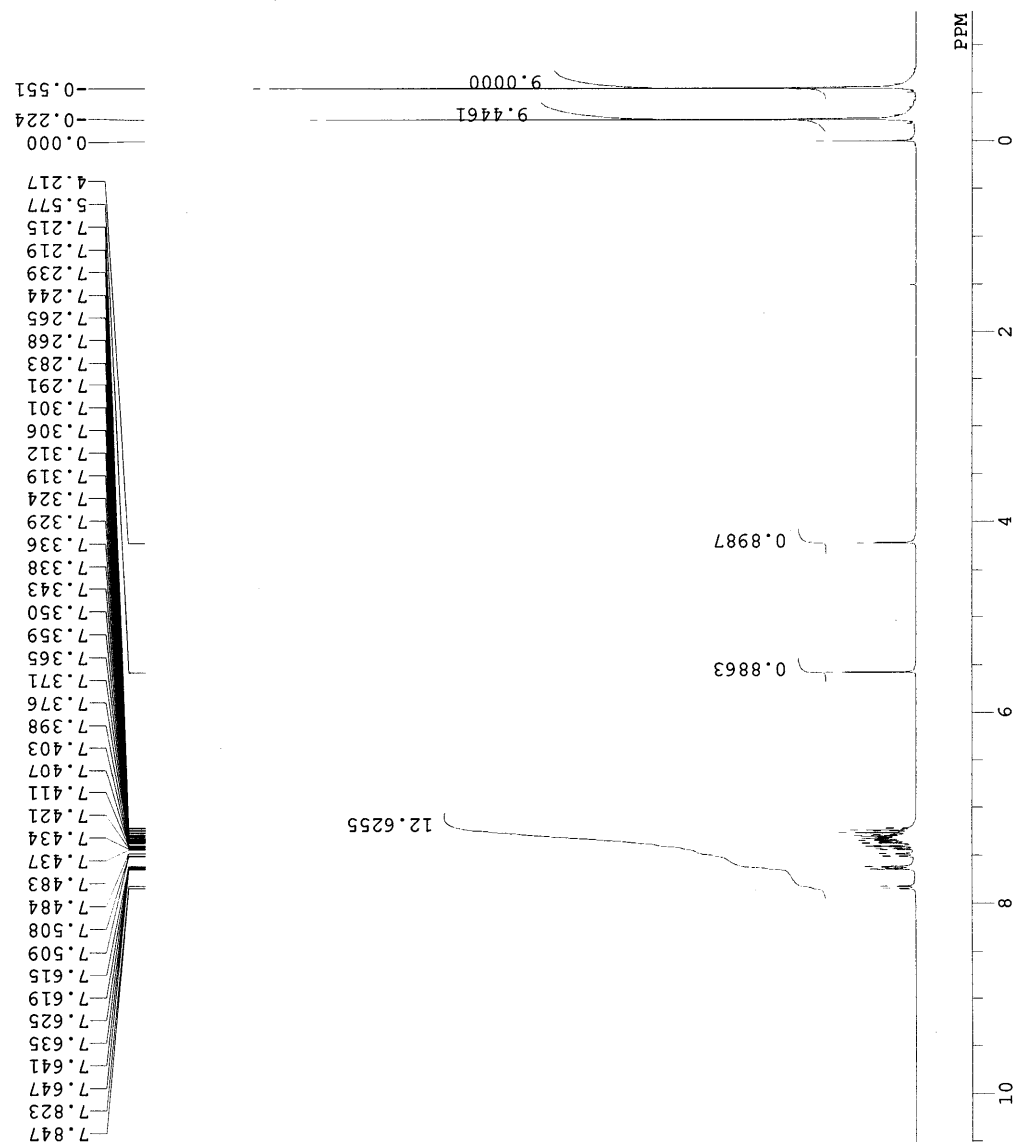
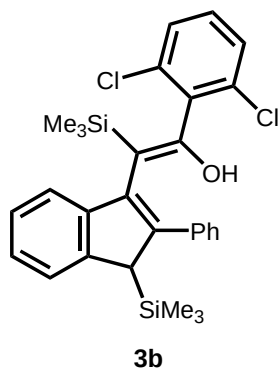


3b-HNMR-withTMS

D:\Huqiaoshu\508\H-TMS-Clea
 R508-H-TMS
 Fri Jan 12 20:35:54 2007
 1H
 NON

300.40 MHz
 130.00 KHz
 1150.0 Hz
 32768
 6013.2 Hz
 16
 5.449 sec
 1.551 sec
 5.8 us
 1H
 24.5 c
 CDCL3
 0.00 ppm
 0.09 Hz
 15
 RGAIN

DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFRO
 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN




```

C:\WINNR99\COMMON\DEFAULT
R508-H-TMS
FRI JUN 12 20:18:52 2007

DRFILE
COUNT
DSTART
DEND
CHAN 1H
EXMOD MON
300.40 MHz
130.00 KHz
1150.0 Hz
32768
6013.2 Hz
16
5.449 sec
1.551 sec
5.8 us
1H
IRNUC
CTEMP
CDOL3
SLVNT
EXREF
BT
RGAIN
0.00 ppm
0.09 Hz
13

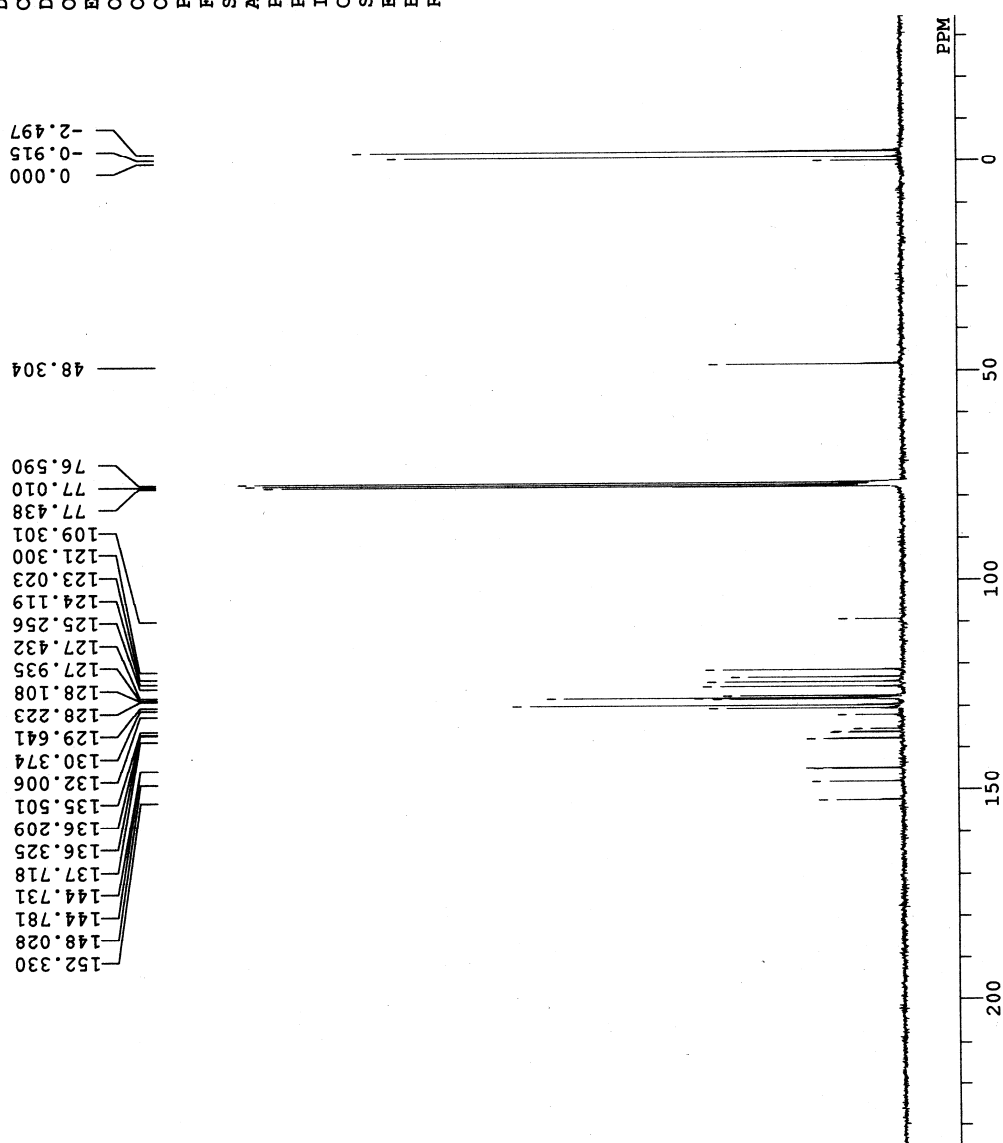
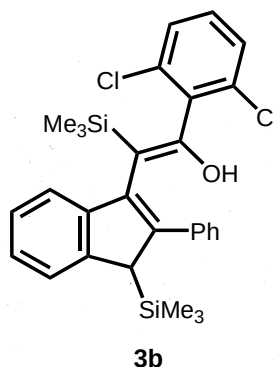
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3b-CNMR-withTMS

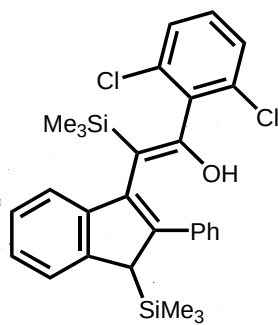
D:\Huqiaoshu\508\C-TMS.als
 COMNT
 DATIM Fri Nov 24 12:18:26 2006
 OBNUC 13C
 EXMOD BCM

OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 2240
 ACQTM 1.506 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 24.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 26

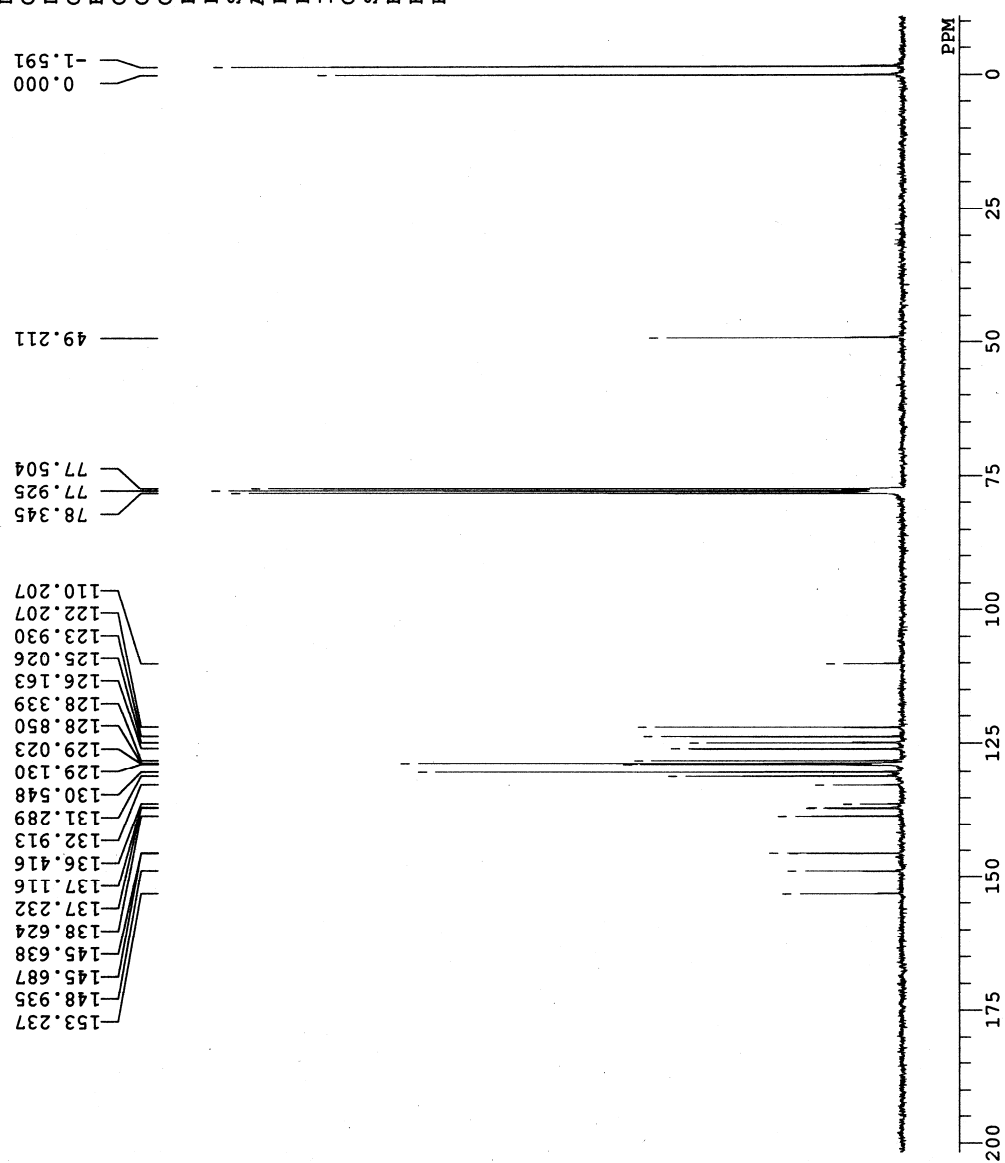


3b-CNMR-no-TMS

D:\Hugiaoshu\508\C-NO-TMS-
 DFIL E Fri Nov 24 10:16:28 2006
 COMNT 13C
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFRQ 124.00 KHz
 OBSET 1840.0 Hz
 OBFIN 32768
 POINT 20408.1 Hz
 FREQU 1473
 SCANS 1.606 sec
 ACQTM 1.394 sec
 PD 5.0 us
 PW1 1H
 IRNUC 24.0 c
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.62 Hz
 BF 27
 RGAIN

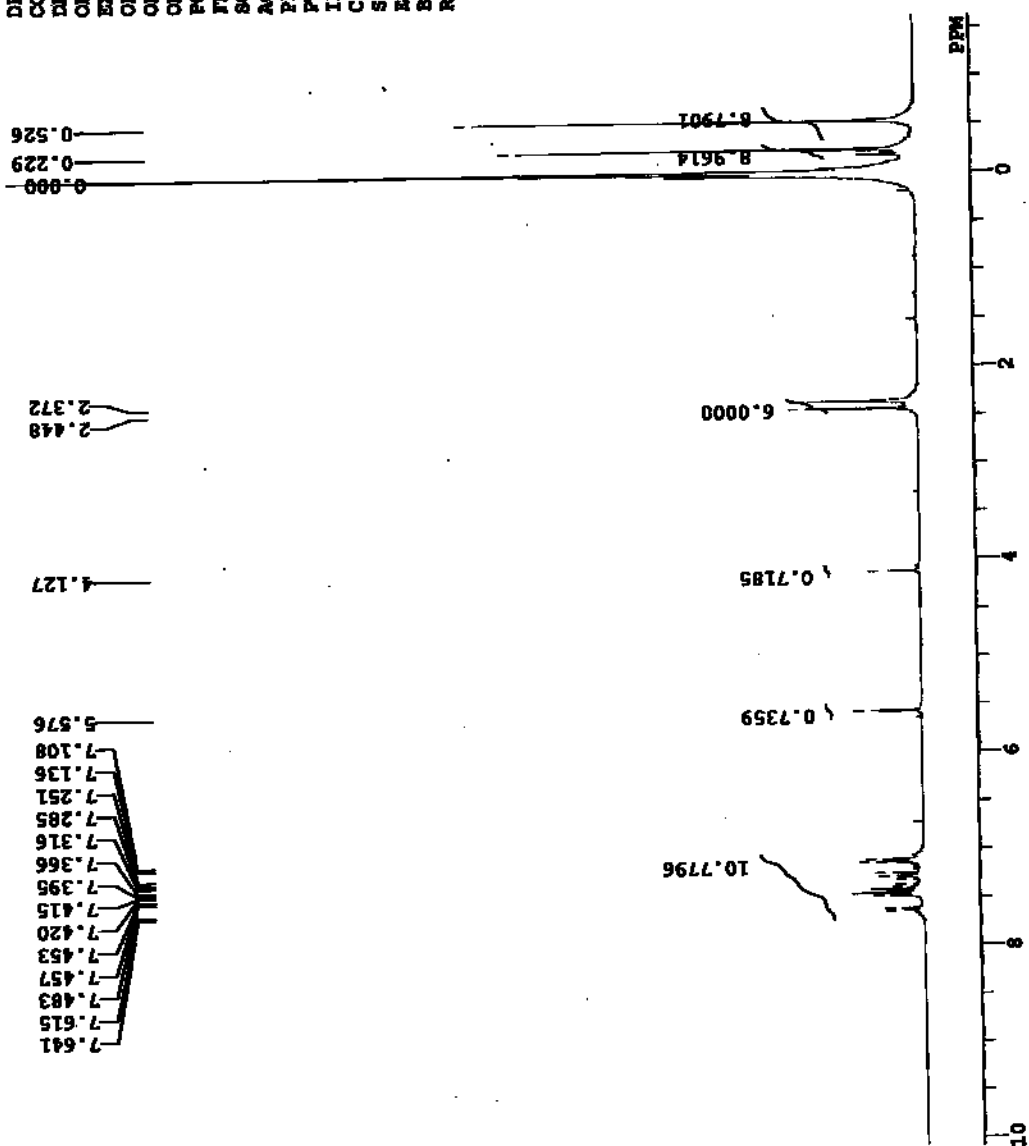
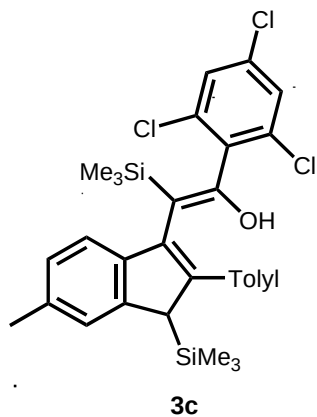


3b



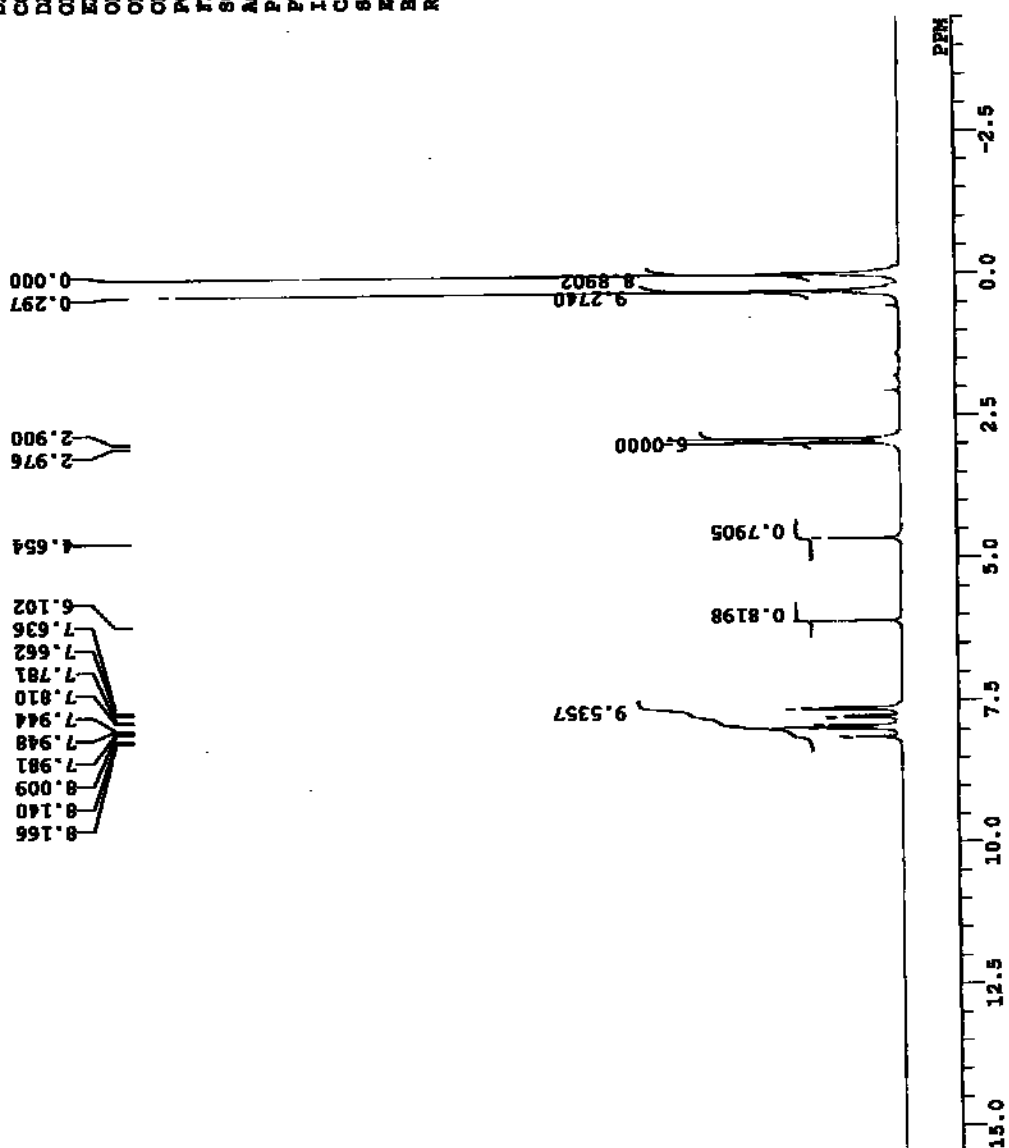
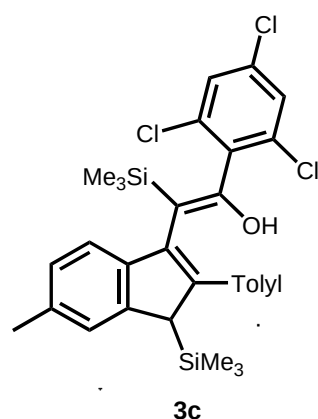
3c-HNMR-withTMS

D:\LDS\132\TMS-H.mls
 Mon Jan 08 18:35:23 2007
 1H
 300.40 MHz
 130.00 MHz
 1150.0 Hz
 32768
 6013.2 Hz
 16
 5.449 sec
 1.551 sec
 5.8 us
 1H
 23.9 c
 CDCl3
 0.00 ppm
 0.09 Hz
 15

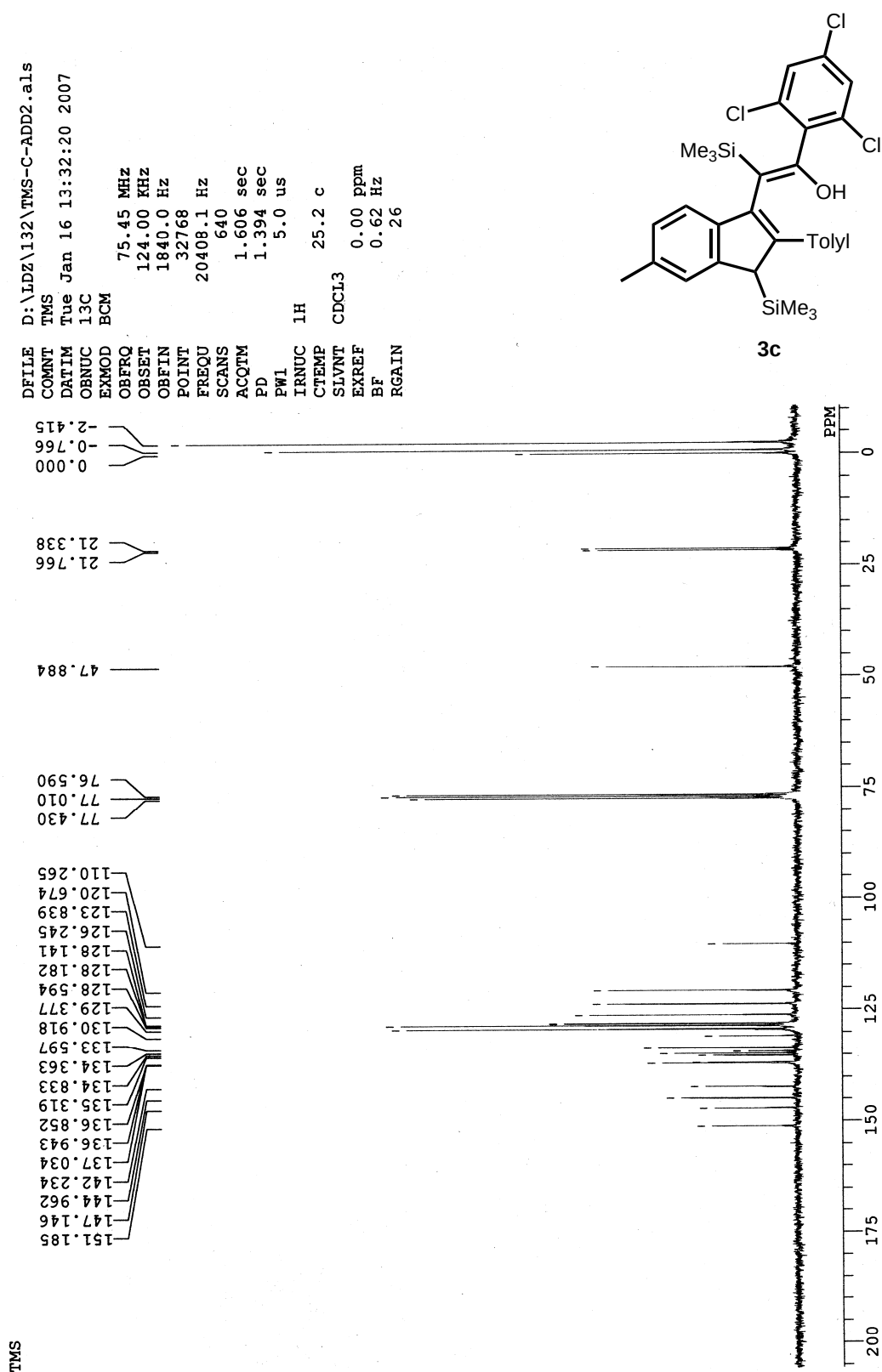


3c-HNMR-no-TMS

D:\LDS\132\Clear H.als
 DATE Mon Jan 08 09:45:44 2007
 COMPT 1H
 EXMOD NON
 OBPRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQ 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.851 sec
 PW1 5.6 us
 1H
 INOC 23.3 c
 CDCL3
 STMT 0.00 ppm
 EXREF 0.09 Hz
 RF 17
 RGAIN

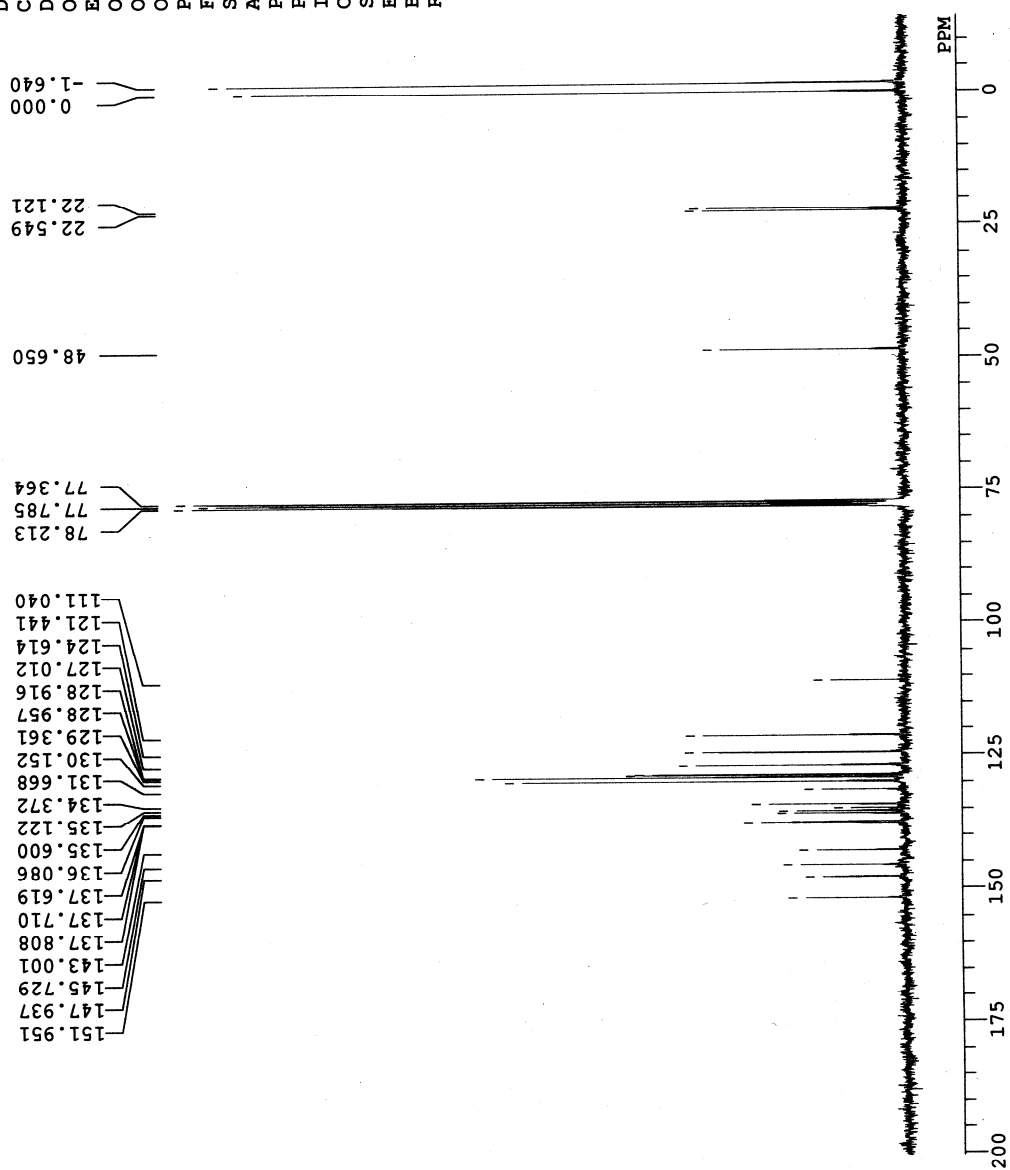
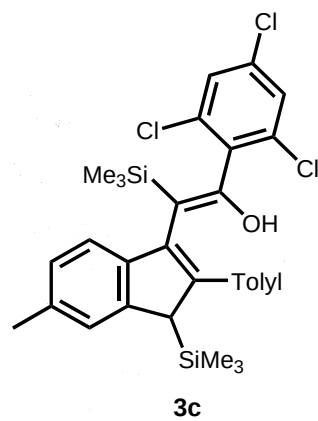


3c-CNMR-withTMS



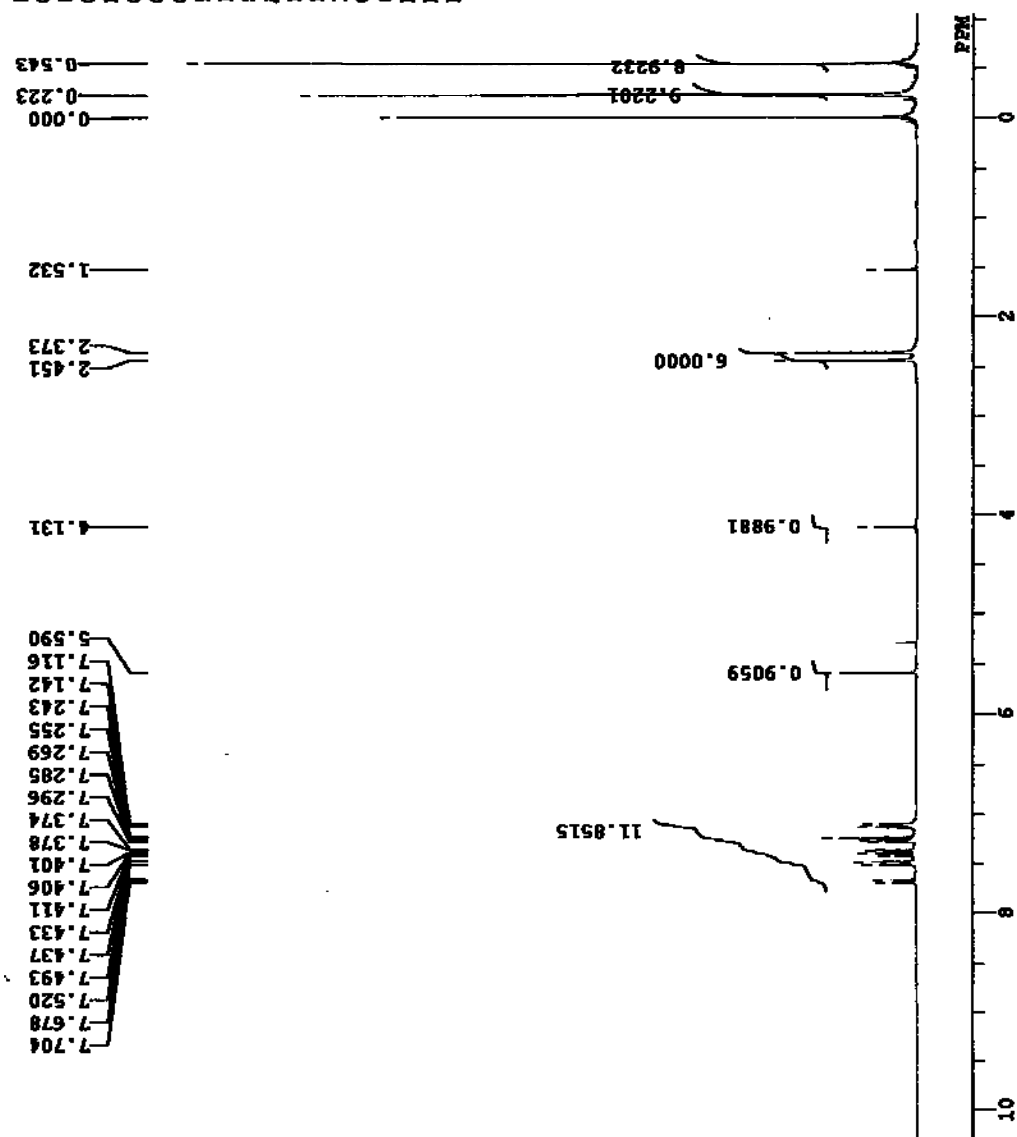
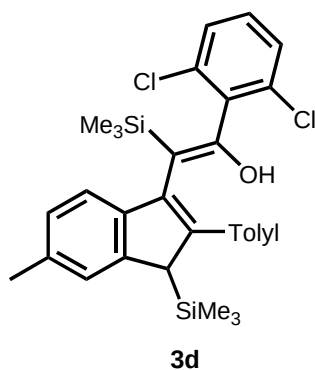
3c-CNMR-no-TMS

D:\LDZ\132\clear c2.als
 COMNT Mon Jan 08 10:36:44 2007
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFREQ 124.00 KHz
 OBSET 1840.0 Hz
 OBFIN 32768
 POINT 20408.1 Hz
 FREQU 952
 SCANS 1.606 sec
 ACQTM 1.394 sec
 PD 5.0 us
 IRNUC 1H
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 27



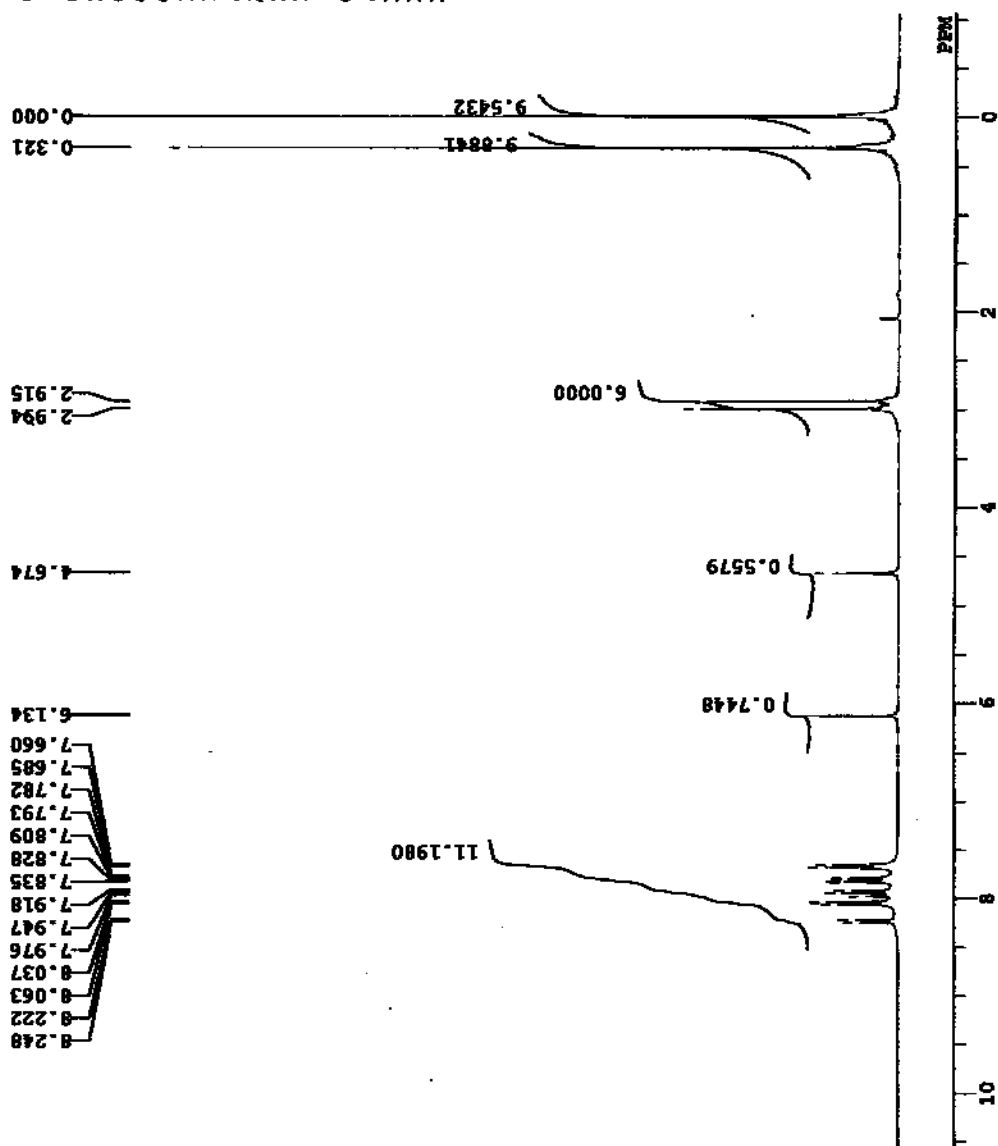
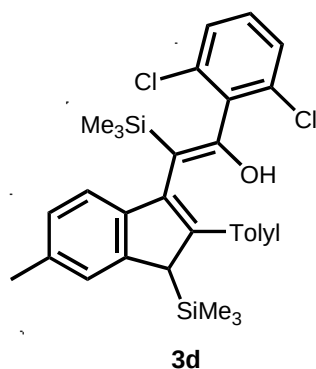
3d-HNMR-withTMS

D:\LDZ\139\clear-h-tms.als
 DATE: Fri Jan 12 12:43:04 2007
 1H
 300.40 MHz
 130.00 KHz
 1150.0 Hz
 32768
 6013.2 Hz
 16
 5.449 sec
 1.551 sec
 5.8 us
 1H
 24.1 C
 CDCL3
 0.00 ppm
 0.09 Hz
 20



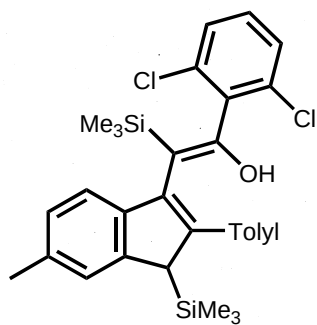
3d-HNMR-no-TMS

D:\LD2\139\clear -h.als
 COMET F11 Jan 12 10:57:21 2007
 CHNOC 1H
 EXMOD NOX
 OBSFQ 300.40 MHz
 OBSFQ 130.00 MHz
 OBSFQ 1150.0 Hz
 POINT 32768
 FREQ 6013.2 Hz
 SCANS 16
 ACQIM 5.449 sec
 PD 1.551 sec
 PWL 5.8 us
 1H
 23.9 C
 CDCL3
 0.00 ppm
 0.09 Hz
 18
 RGAIN

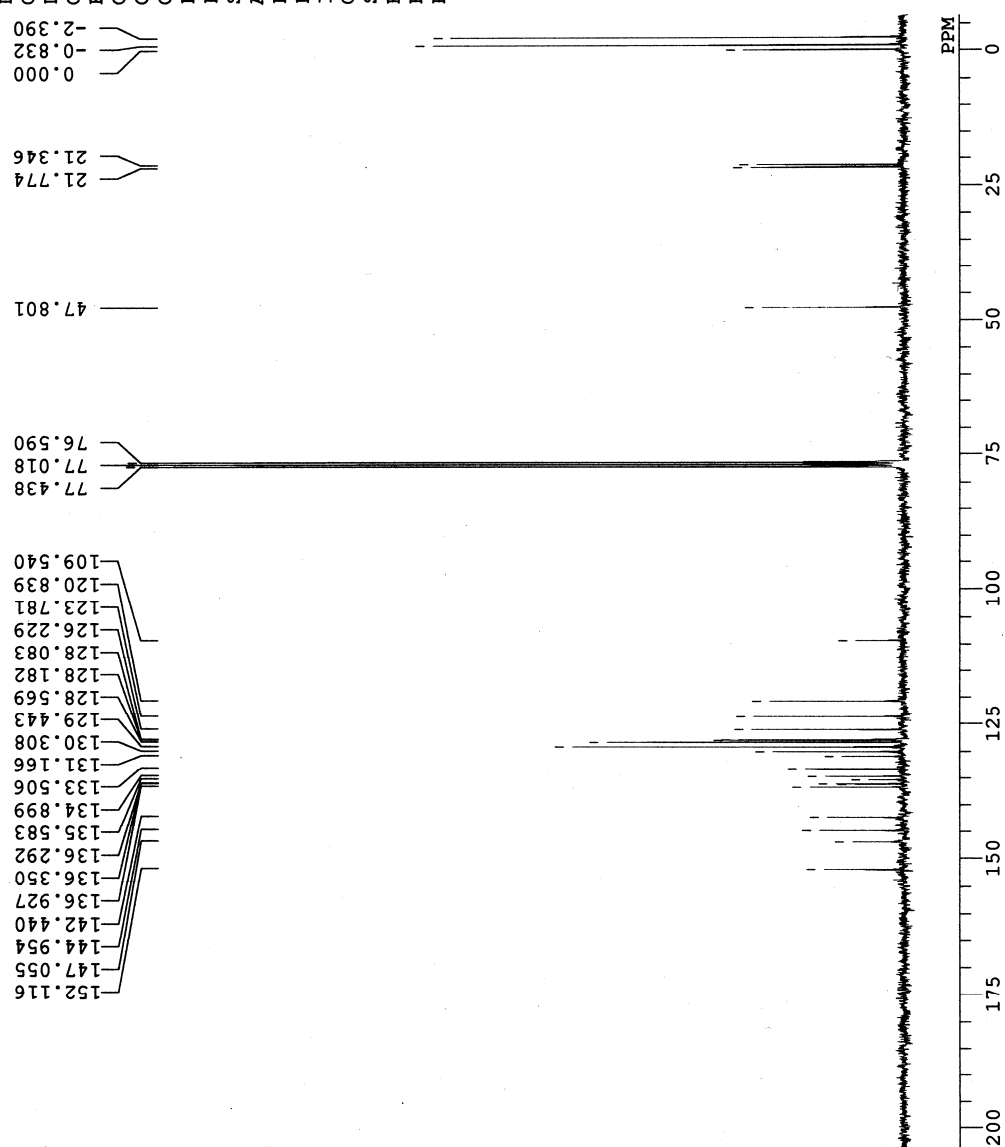


3d-CNMR-withTMS

D:\LDZ\139\clear -c-tms-2
 DFILE COMNT
 DATIM Fri Jan 12 12:32:03 2007
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 720
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 24.5 c
 SLVNT CDCl3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 28



3d



3d-CNMR-no-TMS

D:\LDZ\139\clear -c-smooth2
 COMNT Fri Jan 12 10:46:40 2007
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFRQ 124.00 KHz
 OBSET 1840.0 Hz
 OBFIN 32768
 POINT 20408.1 Hz
 FREQU 480
 SCANS 1.606 sec
 ACQTM 1.394 sec
 PD 5.0 us
 PW1 1H
 IRNUC 24.5 c
 CTMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.62 Hz
 BF 26
 RGAIN

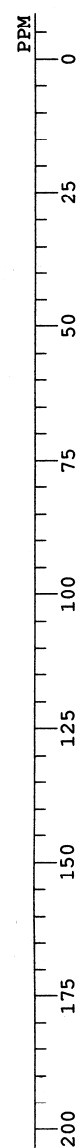
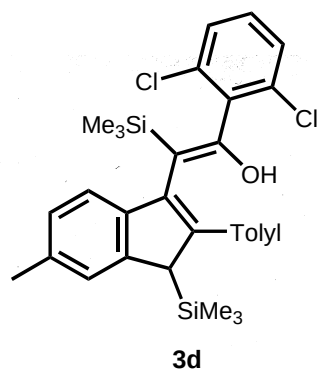
0.000
 -1.566

22.607
 22.170

48.634

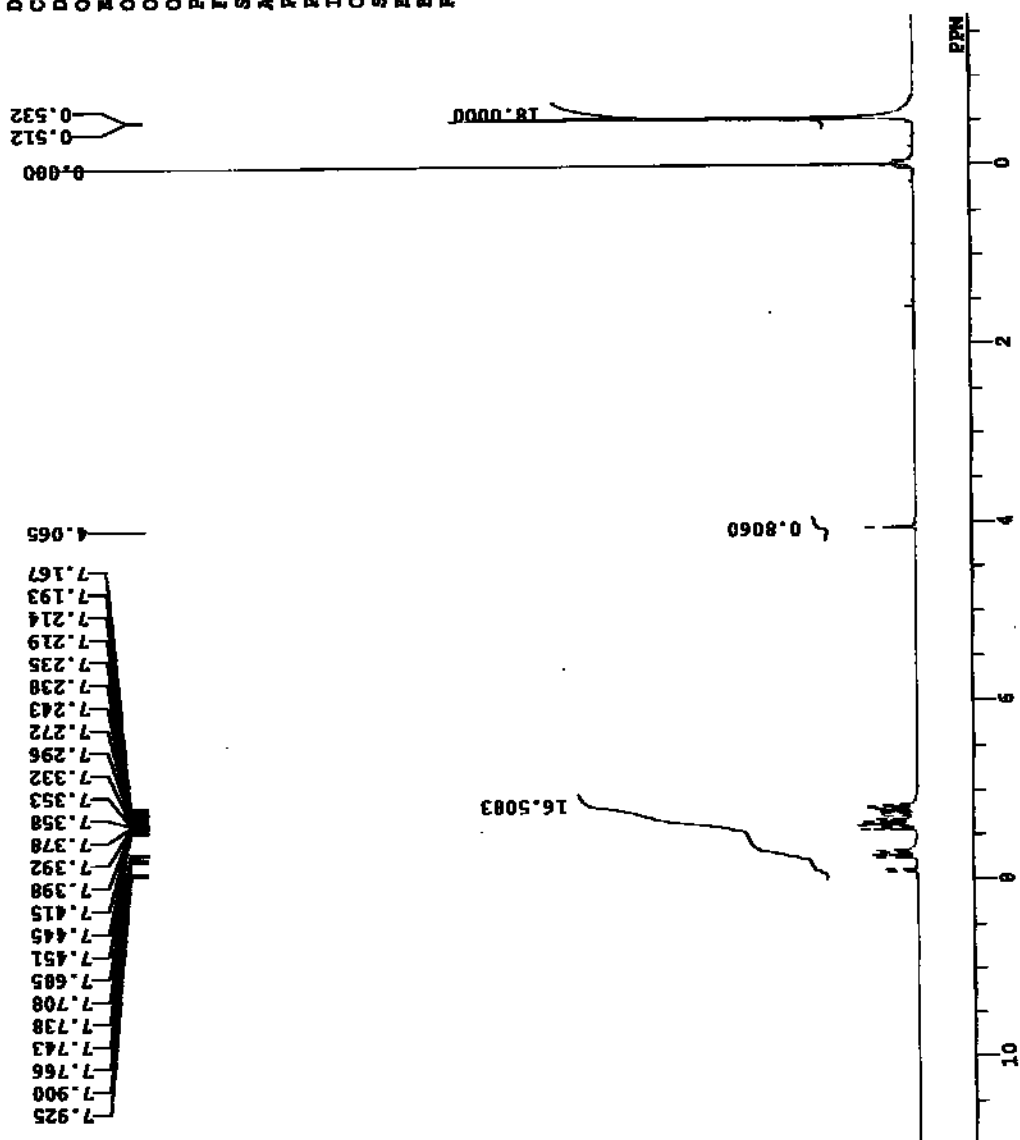
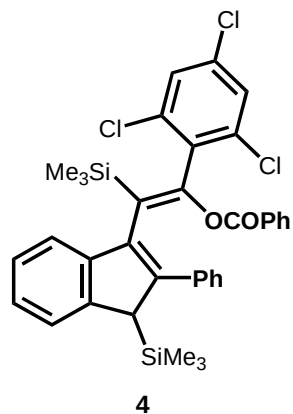
78.271
 77.842
 77.422

152.948
 147.880
 145.786
 143.273
 137.759
 137.174
 137.124
 136.416
 135.723
 134.339
 131.998
 131.141
 130.276
 129.402
 129.006
 128.916
 127.061
 124.614
 121.671
 110.364



4-HNMR-withTMS

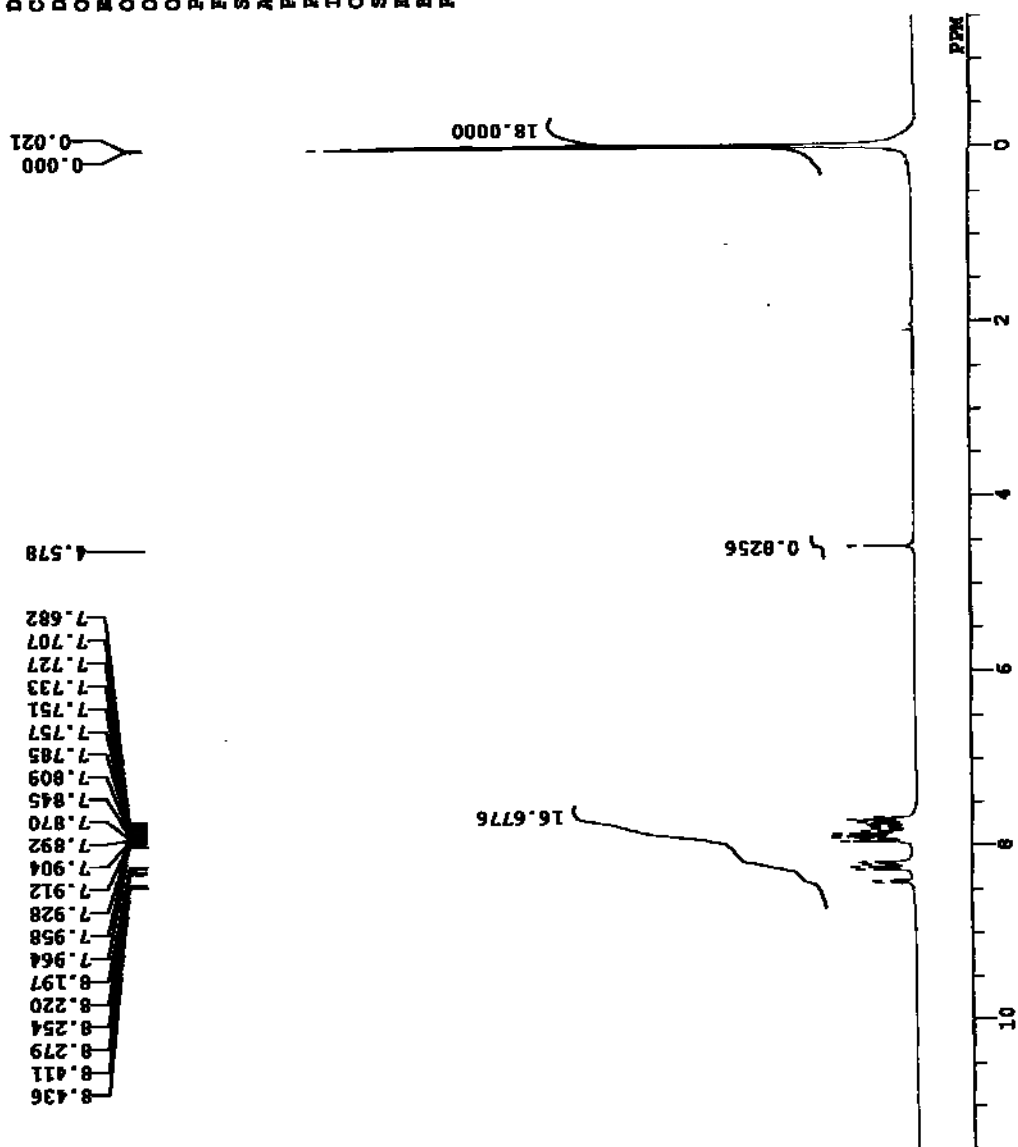
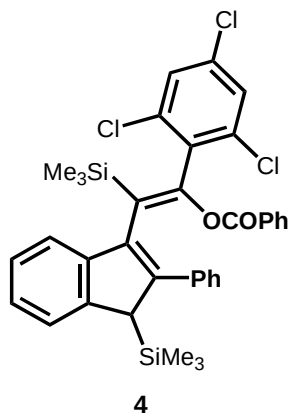
D:\Rugimashu\506\WITH-TMS\H
 COMPT Wed Jan 10 21:26:08 2007
 DATIM 1H
 OBNUC 1H
 EXMOD NON
 OBFSQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 IRNUC 1H
 CTMP 23.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 RF 0.09 Hz
 RGAIN 14



4-HNMR-no-TMS

D:\Ruqiaoshu\506\NO-TMS\4-2

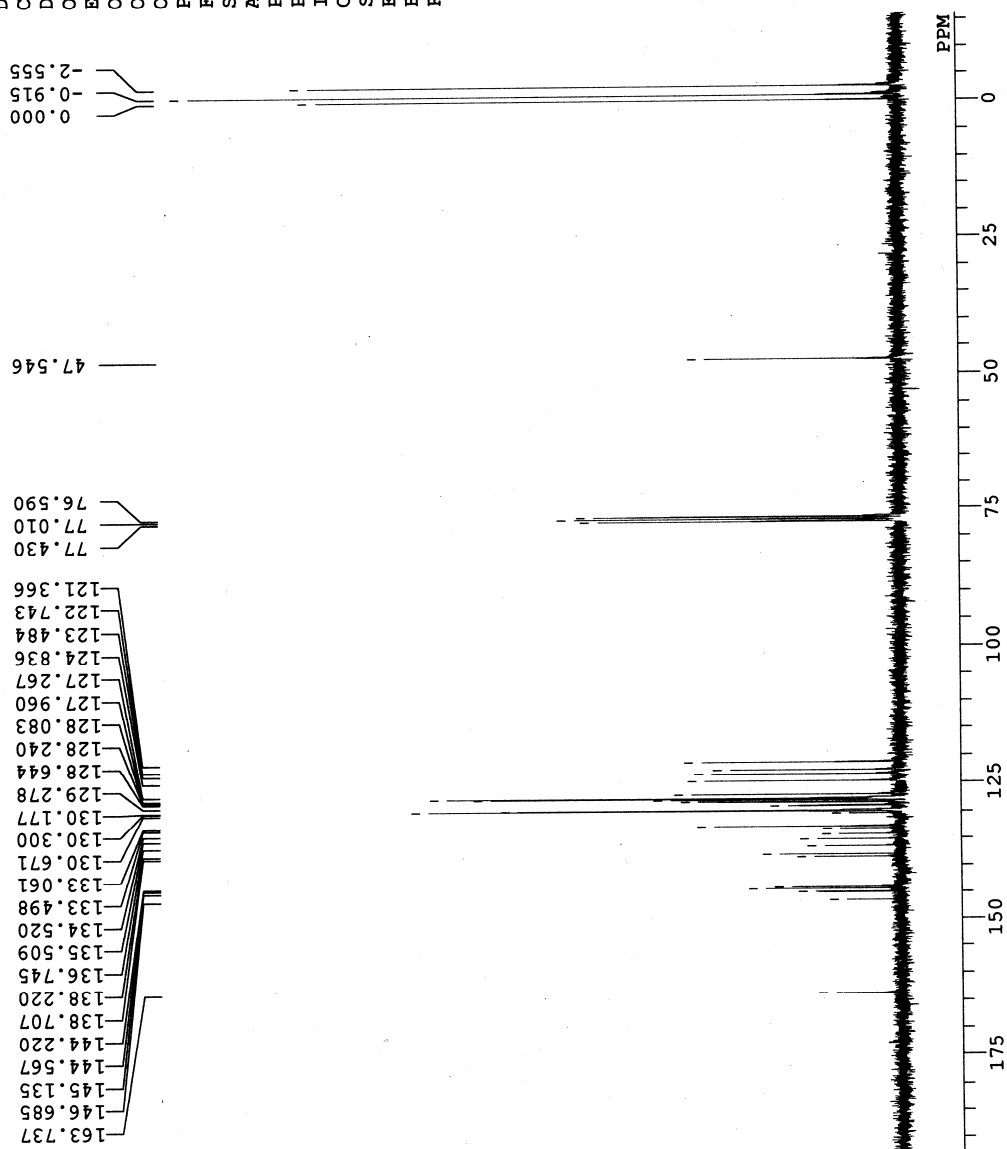
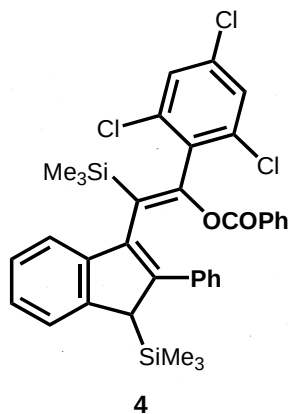
DATE Wed Jan 10 20:43:05 2007
 INSTR 1H
 EXMOD WOH
 OBSFQ 300.40 MHz
 OBSFQ 130.00 MHz
 OBSFQ 1150.0 Hz
 POINT 32768
 FREQ 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.8 us
 INOC 1H
 CTMP 23.3 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 RF 0.62 Hz
 RGAIN 14



4-CNMR-withTMS

C:\WINNMR95\COMMON_DEFAULT

DFILE COMNT Wed Jan 10 21:19:16 2007
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBPRQ 124.00 KHz
 OBSET 1840.0 Hz
 OFIN 32768
 POINT 20408.1 Hz
 FREQU 401
 SCANS 1.606 sec
 ACQTM 1.394 sec
 PD 5.0 us
 PW1 1H
 IRNUC 24.1 C
 CTMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.62 Hz
 BF 26
 RGAIN



4-CNMR-no-TMS

D:\Hugiaoshu\506\NO-TMS\C-4

COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWL
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

0.000
-1.632

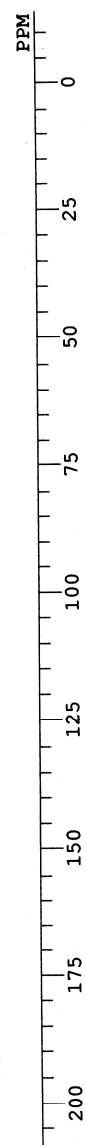
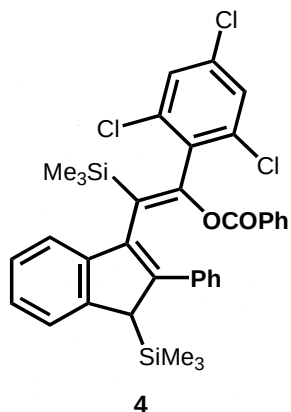
48.461

164.660
147.591
146.050
145.481
145.135
139.622
139.127
137.660
136.424
135.427
134.405
133.984
131.594
131.215
131.092
130.185
129.559
129.163
129.006
128.875
128.182
125.759
124.399
123.666
122.281
78.362
77.941
77.513

Wed Jan 10 20:36:09 2007

13C
BCM

75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
400
1.606 sec
1.394 sec
5.0 us
1H
23.9 C
CDCL3
0.00 ppm
0.62 Hz
25



6-HNMR-withTMS

D:\Huqiaoshu\502\H-2.sls

COMET Thu Nov 23 18:40:58 2006

1H

NON

300.40 MHz

130.00 KHz

1150.0 Hz

32768

6013.2 Hz

8

5.449 sec

1.551 sec

5.8 us

1H

24.1 °C

CDCL3

0.00 ppm

0.09 Hz

14

DFILE

COMET

DATIM

1H

NON

OBFRQ

OBSET

OBFIN

POINT

FREQU

SCANS

ACQTM

PD

FWL

IRNUC

CTEMP

SLVNT

EXREF

BF

RGAIN

0.000

0.305

7.915
7.910
7.886
7.716
7.691
7.523
7.498
7.464
7.457
7.426
7.420
7.375
7.352
7.346
7.338
7.326
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7.250
7.225
5.607
4.131
4.056
3.870
3.795

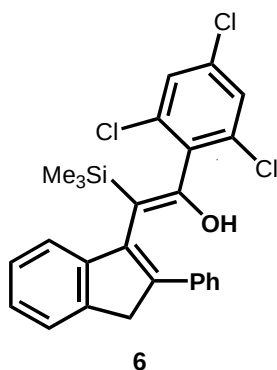
11.2708

0.7646

1.9482

9.0000

PPM
12.5
10.0
7.5
5.0
2.5
0.0
-2.5
-5.0

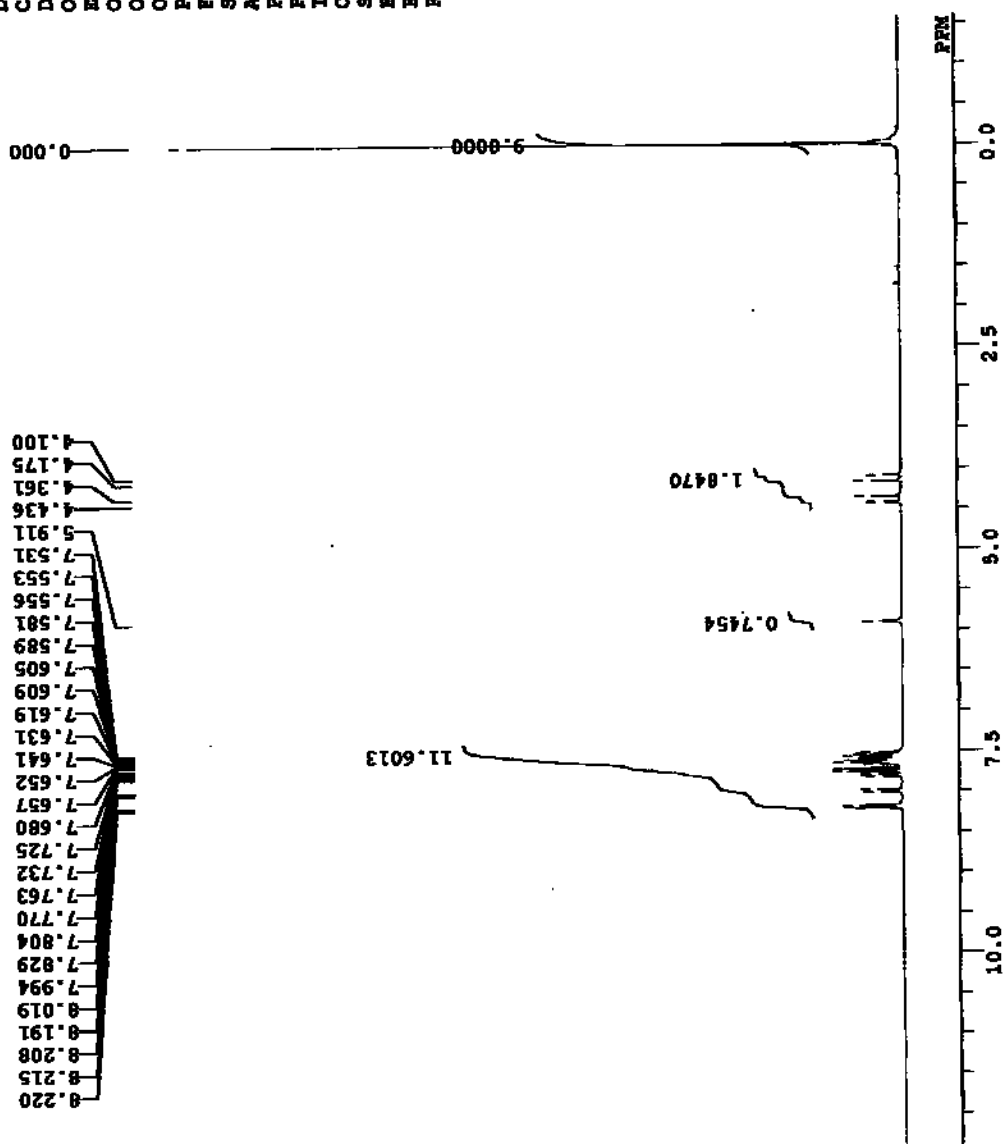
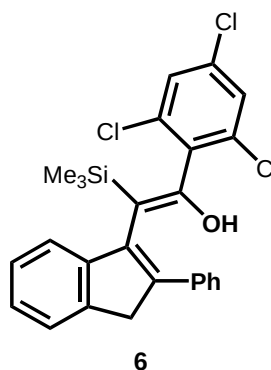


6

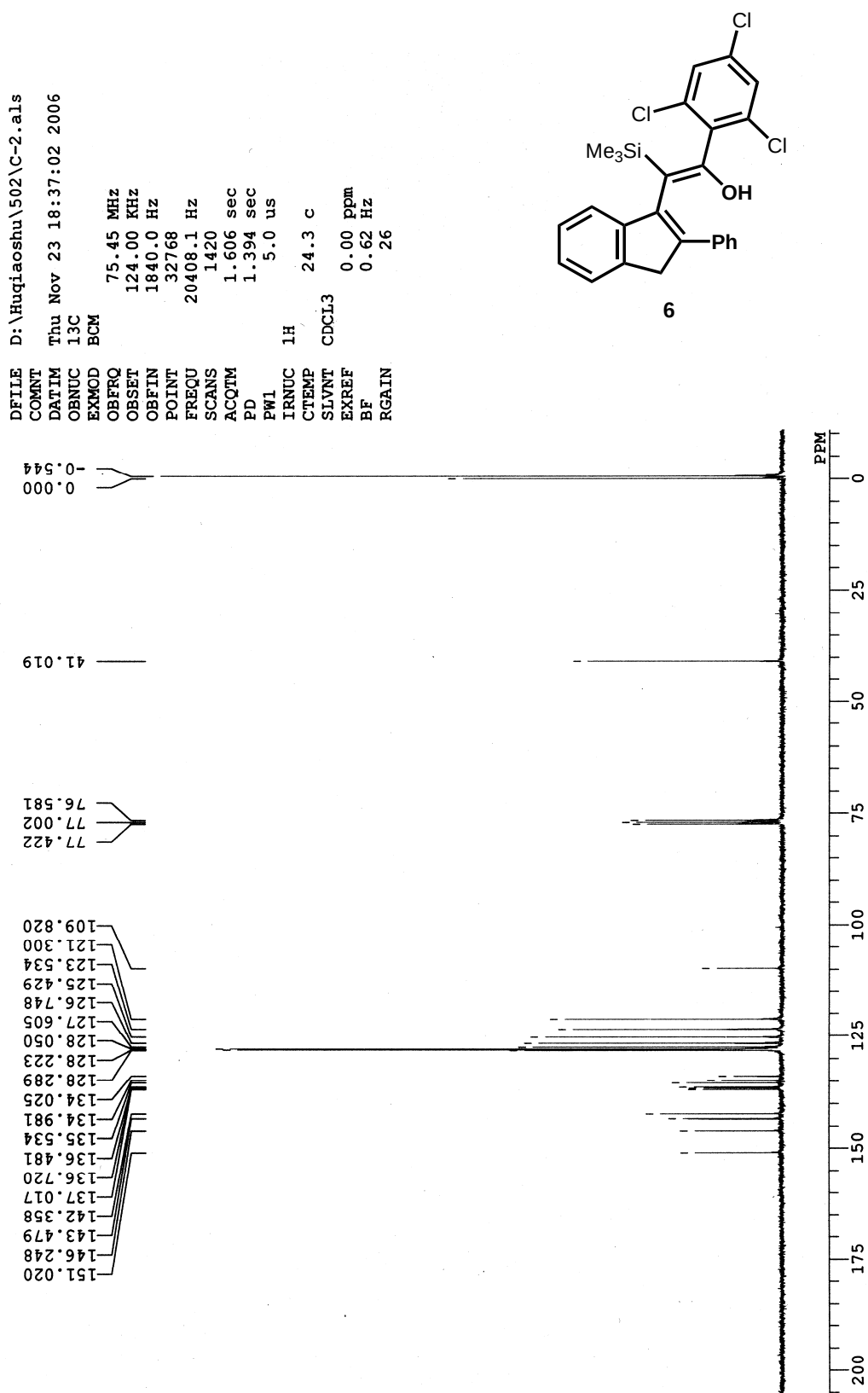
6-HNMR-no-TMS

D:\Ruqiaoshu\502\H-no-TMS-2

DATE Thu Nov 23 17:20:31 2006
 LHM
 EXMOD NON
 OBSFQ 300.40 MHz
 OBSST 130.00 KHz
 OBSIN 1150.0 Hz
 POINT 32768
 FREQ 6013.2 Hz
 SCANS 8
 ACQTM 5.449 sec
 PD 1.551 sec
 FW 5.8 us
 LHM
 CTMP 24.0 c
 CDCL3
 EXNET 0.00 ppm
 BF 0.09 Hz
 RGAIN 14



6-CNMR-withTMS



6-CNMR-no-TMS

D:\Huqiaoshu\502\C-No-TMS-2

DFILE

COMNT

DATIM

OBNUC

EXMOD

OBFRQ

OBSET

OBFIN

POINT

FREQU

SCANS

ACQTM

PD

PW1

IRNUC

CTEMP

SLVNT

EXREF

BF

RGAIN

Thu Nov 23 17:18:26 2006

13C

BCM

75.45 MHz

124.00 KHz

1840.0 Hz

32768

20408.1 Hz

621

1.606 sec

1.394 sec

5.0 us

1H

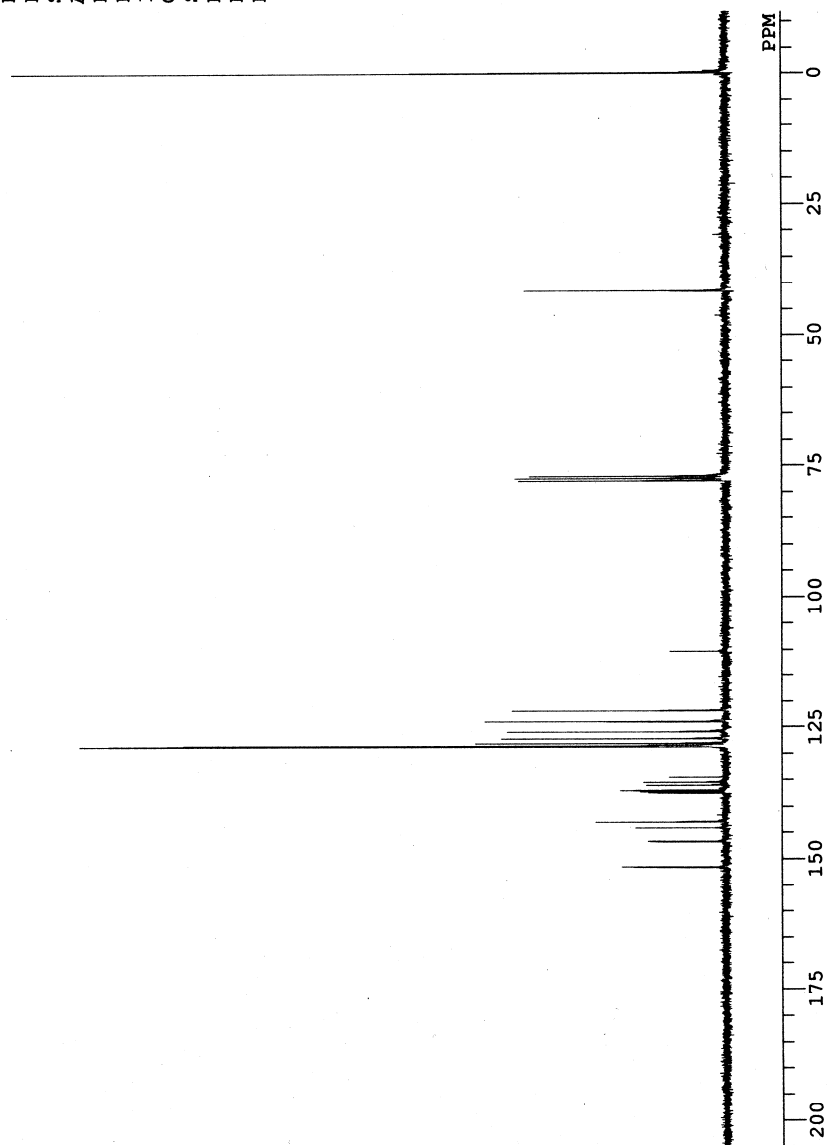
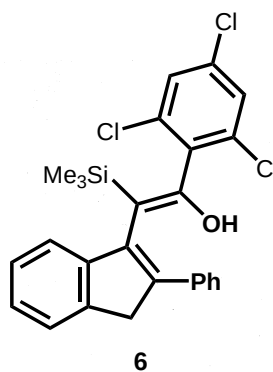
24.3 C

CDCL3

0.00 ppm

0.62 Hz

26



7a-HNMR-withTMS

D:\Hugiaoshu\557\With-TMS\H

Thu Jan 11 22:12:21 2007

1H

MON

300.40 MHz

130.00 KHz

1150.0 Hz

32768

6013.2 Hz

16

5.449 sec

1.551 sec

5.8 us

1H

24.3 C

CDCL3

0.00 ppm

0.09 Hz

15

DTFLE

COMET

DATIM

OBNUC

EXNOU

OBFRQ

OBSET

OBFIN

FOINT

FREQU

SCANS

ACQTM

PD

FW1

IRNUC

CTEMP

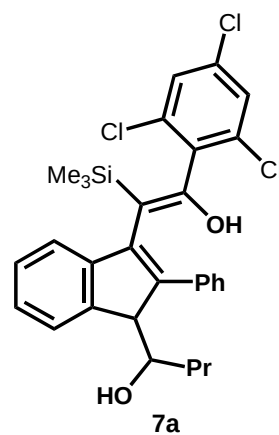
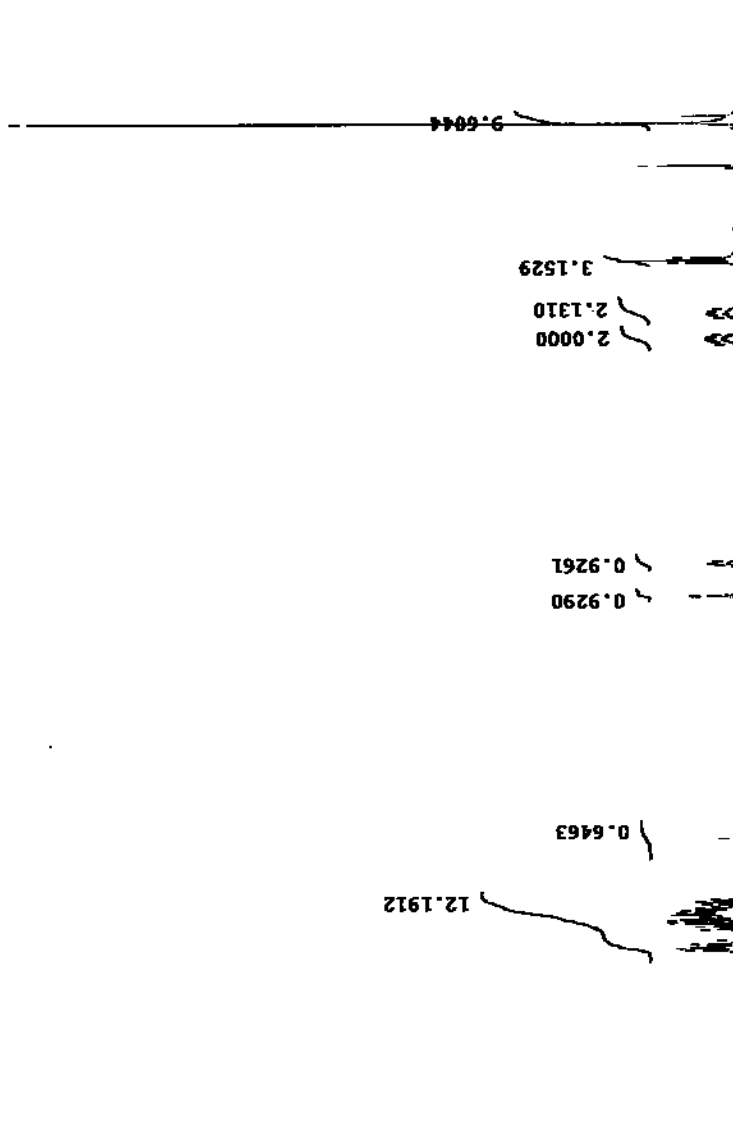
SLVNT

EXREF

BF

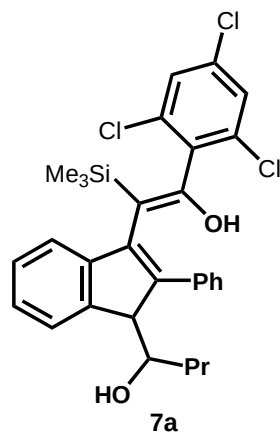
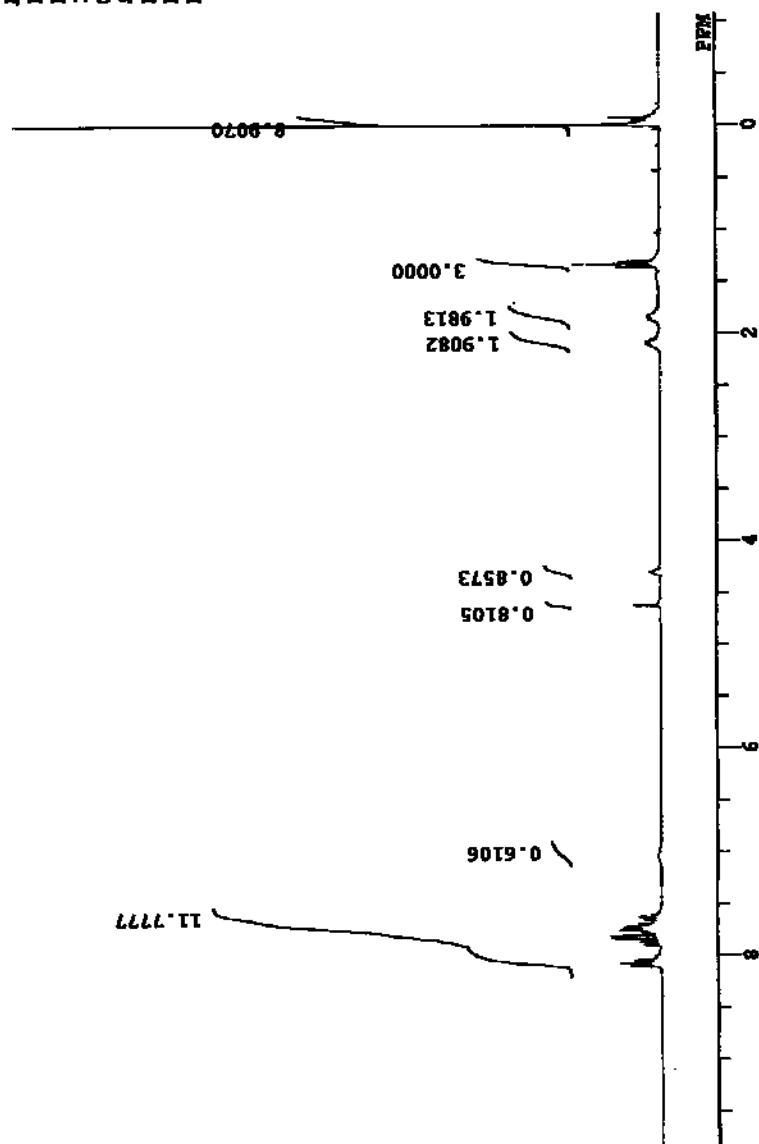
RGAIN

0.415
0.000
0.906
0.930
0.954
1.400
1.425
1.450
1.464
1.489
1.657
1.674
1.684
1.706
1.718
1.729
1.747
3.885
3.903
3.922
4.216
4.224
6.600
7.197
7.222
7.235
7.246
7.264
7.289
7.297
7.312
7.326
7.343
7.351
7.366
7.373
7.379
7.396
7.403
7.413
7.429
7.436
7.445
7.467
7.492
7.598
7.639
7.664
7.675
7.699



7a-HNMR-no-TMS

DFILE C:\WINNMR95\COMMON_DEFAULT
 CONNT Thu Jan 11 21:31:26 2007
 DATIM 1H
 CENUC 1H
 EXMOD NOM
 CBFREQ 300.40 MHz
 CBSET 130.00 MHz
 CBFIN 1150.0 Hz
 POINT 32768
 FREQ 6013.2 Hz
 SCANS 16
 ACQTM 5.449 sec
 PD 1.551 sec
 PW 5.8 us
 1H 24.1 C
 CTEMP CDCL3
 SLVNT CDCL3
 EXRET 0.00 ppm
 BT 0.09 Hz
 RGAIN 14



7a-CNMR-withTMS

D:\Hugiaoshu\557\With-TMS\C

Thu Jan 11 22:08:40 2007

13C

BCM

75.45 MHz

124.00 KHz

1840.0 Hz

32768

20408.1 Hz

401

1.606 sec

1.394 sec

5.0 us

1H

24.9 c

CDCl3

0.00 ppm

0.62 Hz

26

DFILE

COMNT

DATIM

OBNUC

EXMOD

OBFRQ

OBSET

OBFIN

POINT

FREQU

SCANS

ACQTM

PD

PW1

IRNUC

CTEMP

SLVNT

EXREF

BF

RGAIN

0.000

-0.824

14.011

19.689

38.052

56.859

71.991

76.590

77.018

77.438

108.946

121.523

123.995

124.951

127.432

127.605

128.108

128.182

128.207

129.130

134.495

135.262

135.872

136.209

136.696

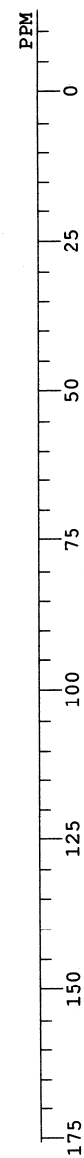
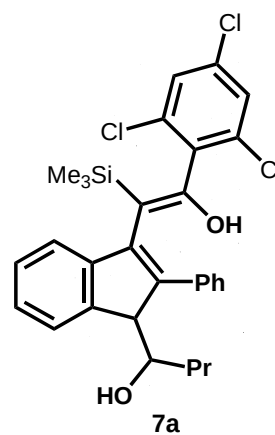
136.819

141.781

146.619

147.393

151.679



7a-CNMR-no-TMS

D:\Huqiaoshu\557\No-TMS\C-C

DFILE

CONNT

DATIM

OBNUC

EXMOD

OBFRQ

OBSET

OBFIN

POINT

FREQU

SCANS

ACQTM

PD

PW1

IRNUC

CTEMP

SLVNT

EXREF

BF

RGAIN

Thu Jan 11 21:25:42 2007

13C

BCM

75.45 MHz

124.00 KHz

1840.0 Hz

32768

20408.1 Hz

657

1.606 sec

1.394 sec

5.0 us

1H

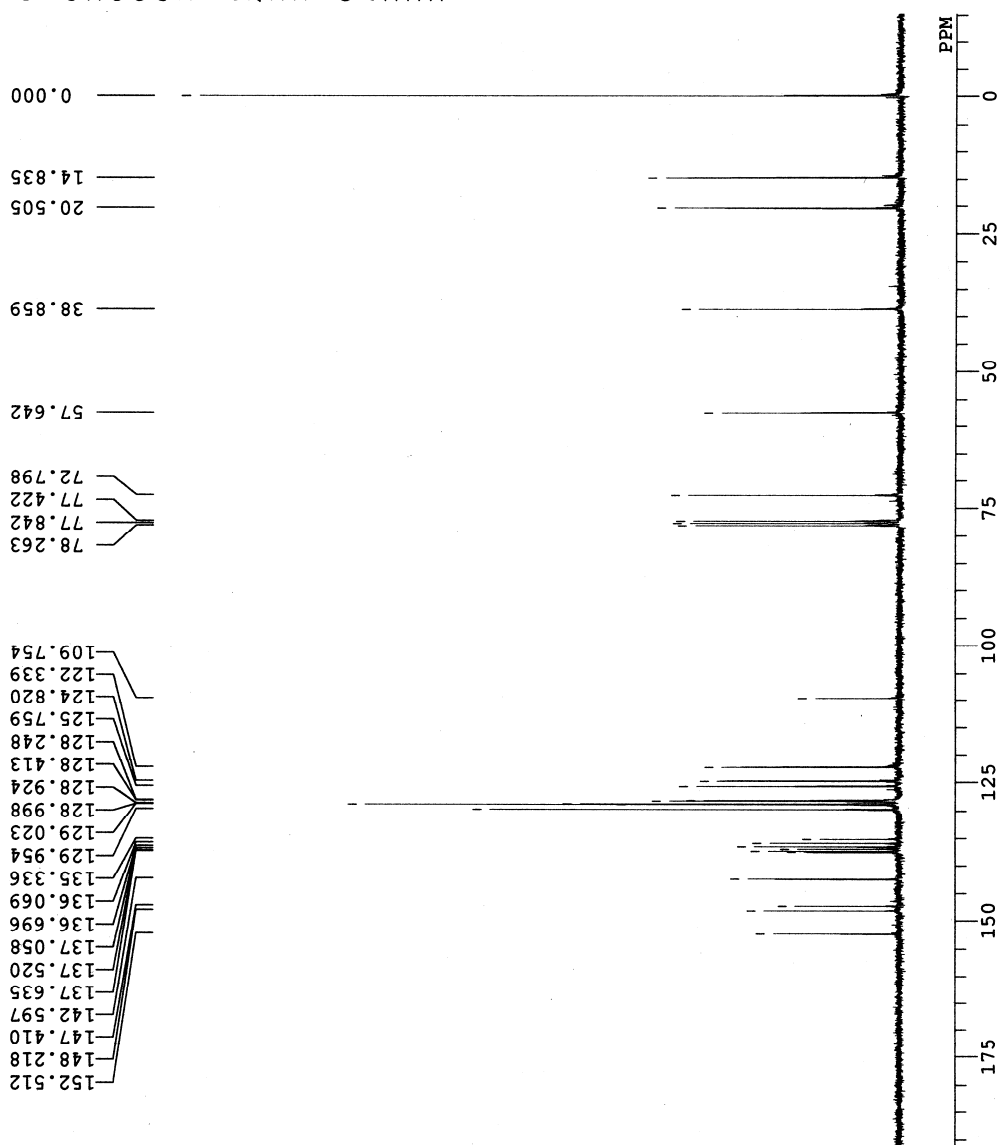
24.7 C

CDCL3

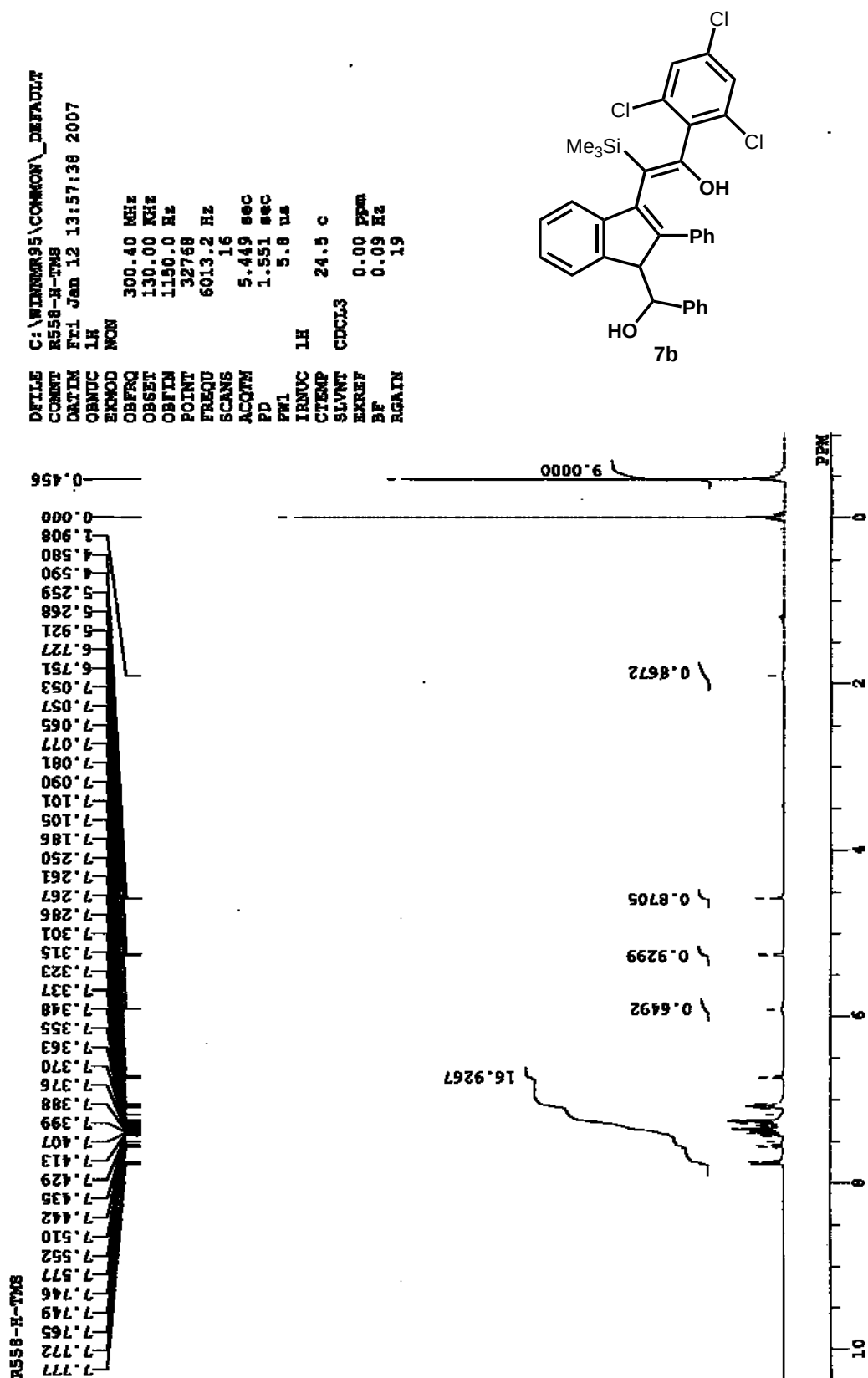
0.00 ppm

0.62 Hz

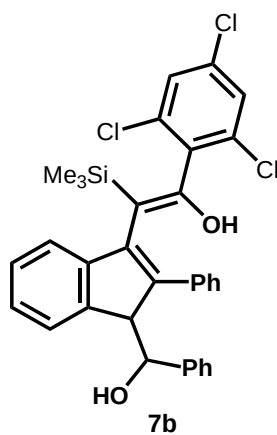
26



7b-HNMR-withTMS



7b-HNMR-no-TMS

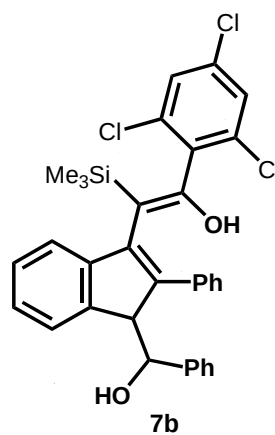


7b-CNMR-withTMS

D:\Huqiaoshu\558\With-TMS\C
R558-C-TMS
Fri Jan 12 13:52:14 2007
OBNUC 13C
EXMOD BCM

75.45 MHz
124.00 KHz
1840.0 Hz
32768
20408.1 Hz
800
1.606 sec
1.394 sec
5.0 us
1H
25.1 c
CDCL3
0.00 ppm
0.62 Hz
26

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

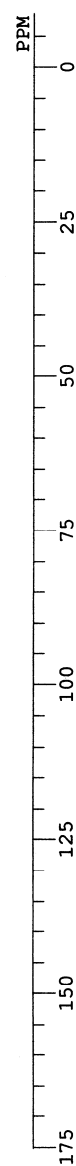


0.000
-0.865

58.474

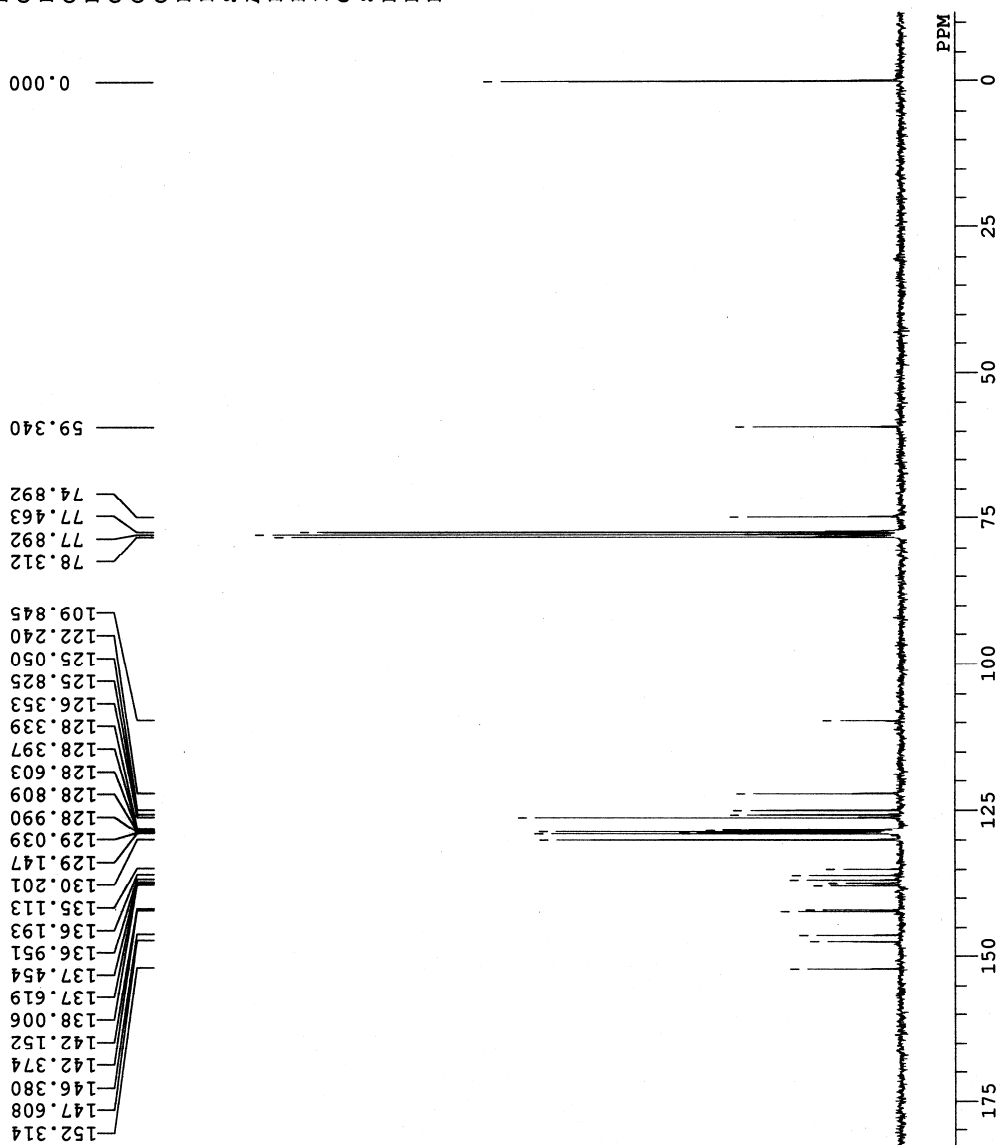
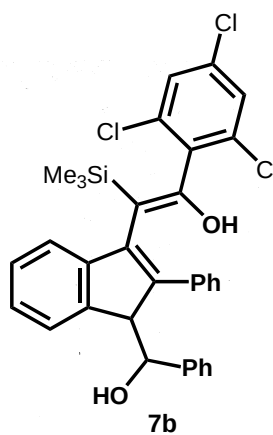
74.026
76.590
77.018
77.438

108.979
121.383
124.185
124.960
125.487
127.473
127.531
127.737
127.943
128.124
128.174
128.281
129.336
134.248
135.328
136.094
136.597
136.762
137.141
141.286
141.517
145.523
146.742
151.457

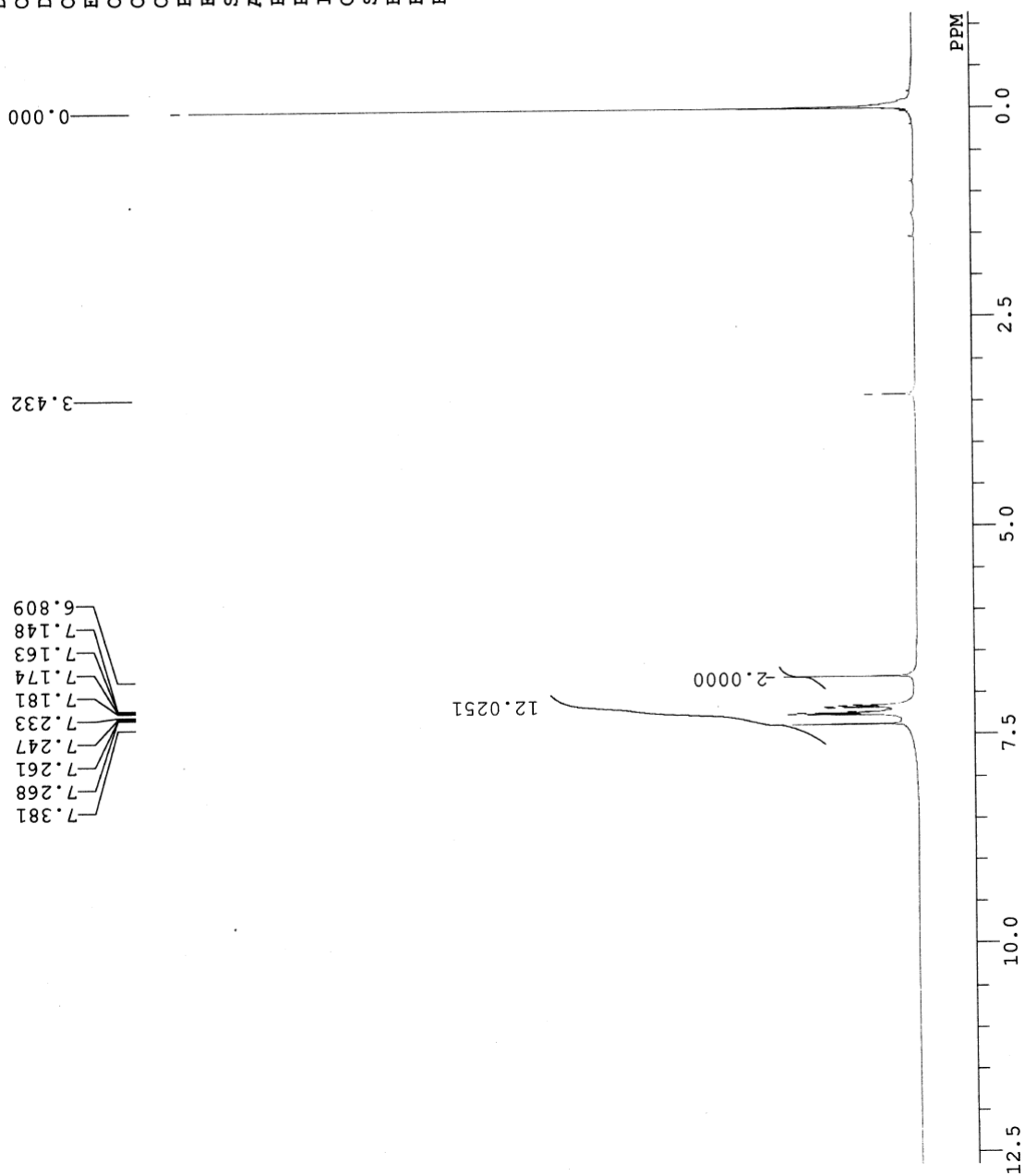
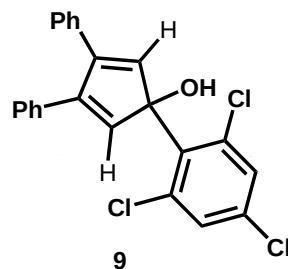


7b-CNMR-no-TMS

D:\Hugiaoshu\558\NotMS\C-c
 R558-C-TMS
 Fri Jan 12 11:46:22 2007
 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 800
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 1H
 IRNUC 24.3 c
 CTMP CDCL3
 SLVNT
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 26

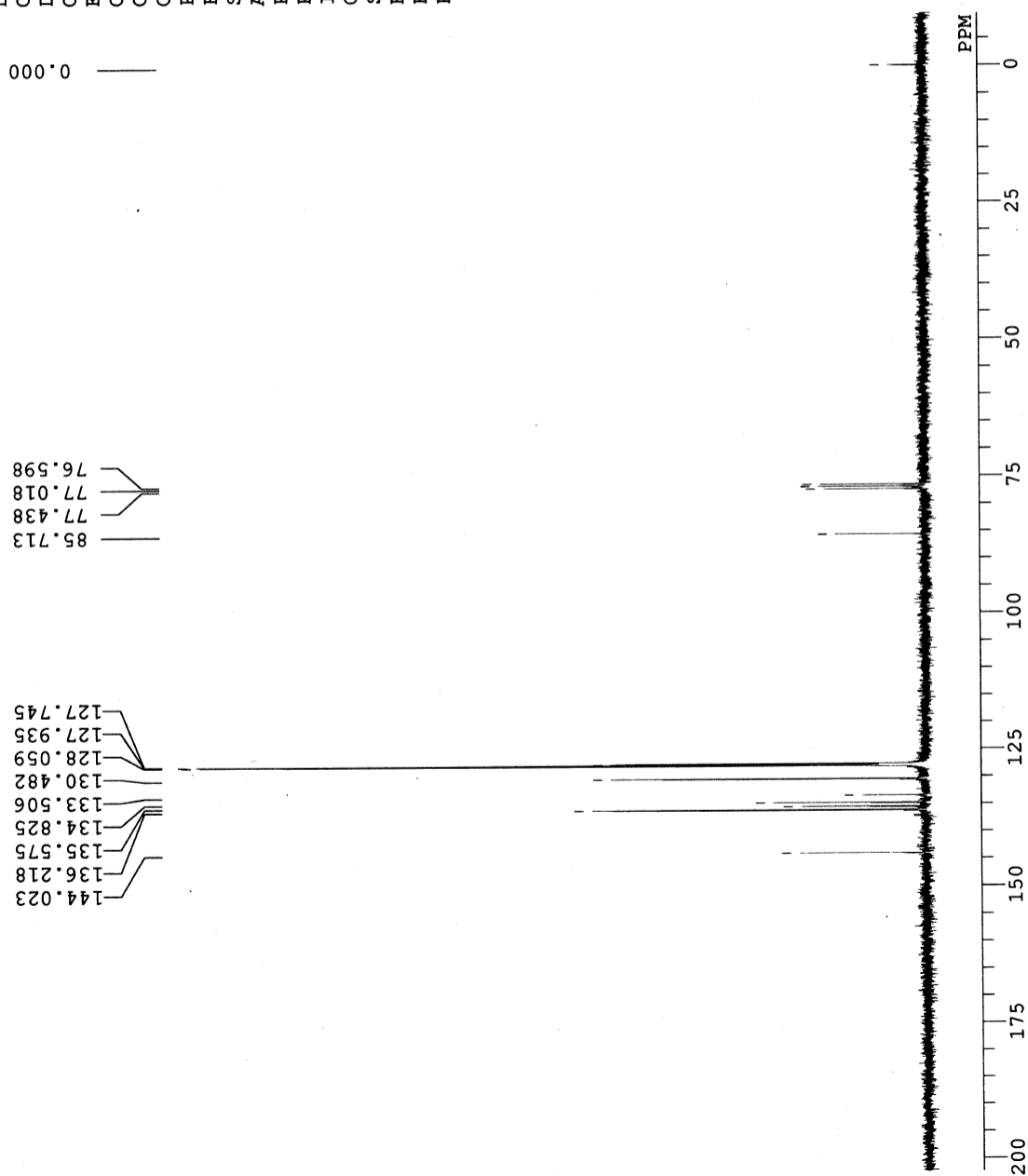
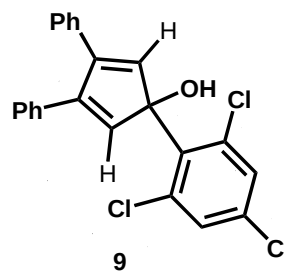


DFILE	D:\LDZ\140\H.als
COMNT	
DATIM	Thu Jan 11 20:08:55 2007
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.0 Hz
POINT	32768
FREQU	6013.2 Hz
SCANS	16
ACQTM	5.449 sec
PD	1.551 sec
PW1	5.8 us
IRNUC	1H
CTEMP	24.1 C
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.09 Hz
RGAIN	16

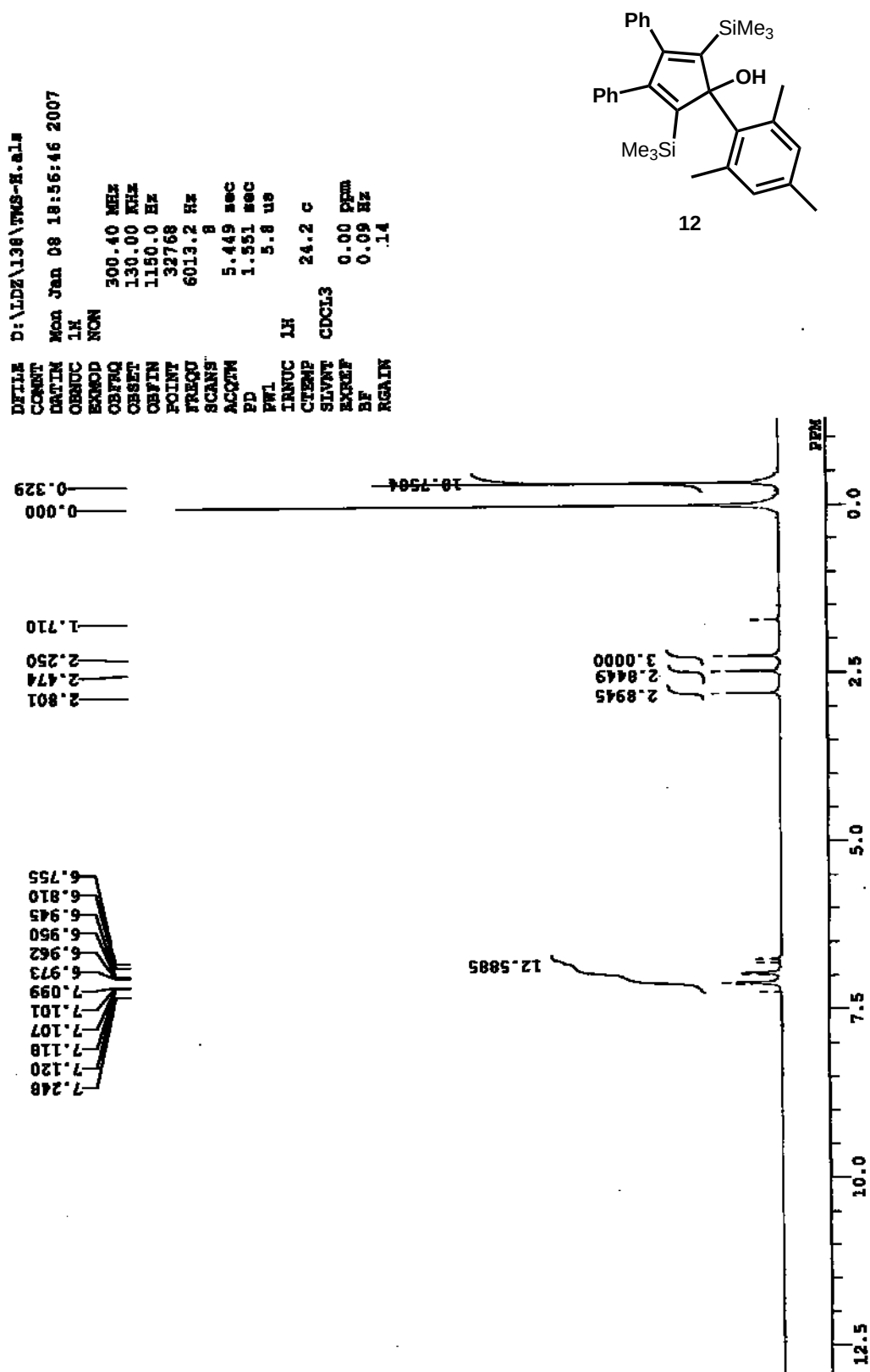


9-CNMR-withTMS

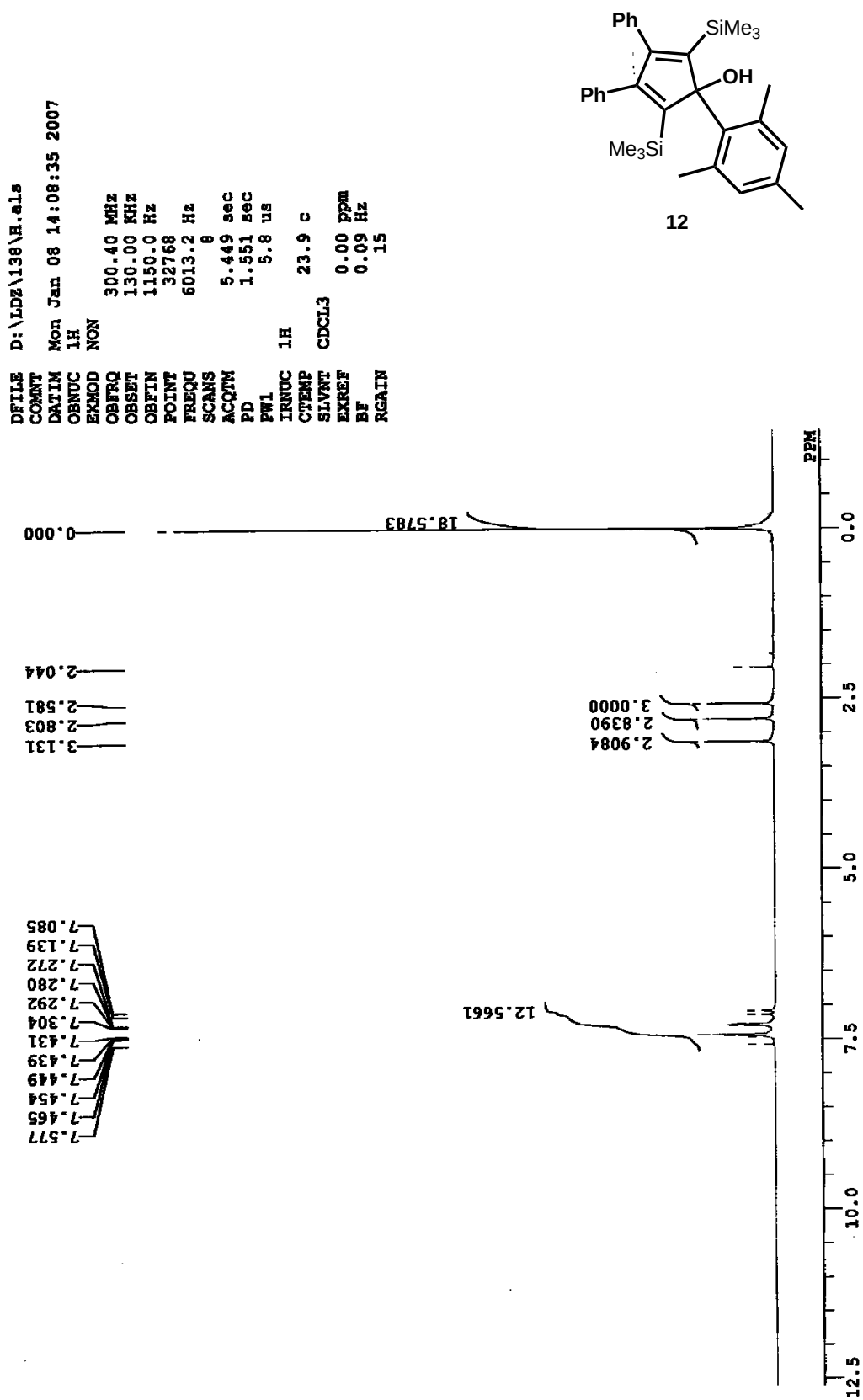
D:\Huqiaoshu\550\C.als
 COMNT Thu Dec 28 21:05:23 2006
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFREQ 124.00 KHz
 OBSET 1840.0 Hz
 OBFIN 32768
 POINT 20408.1 Hz
 FREQU 97
 SCANS 1.606 sec
 ACQTM 1.394 sec
 PD 5.0 us
 PW1 1H
 IRNUC 24.2 C
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.62 Hz
 BF 26
 RGAIN



12-HNMR-withTMS

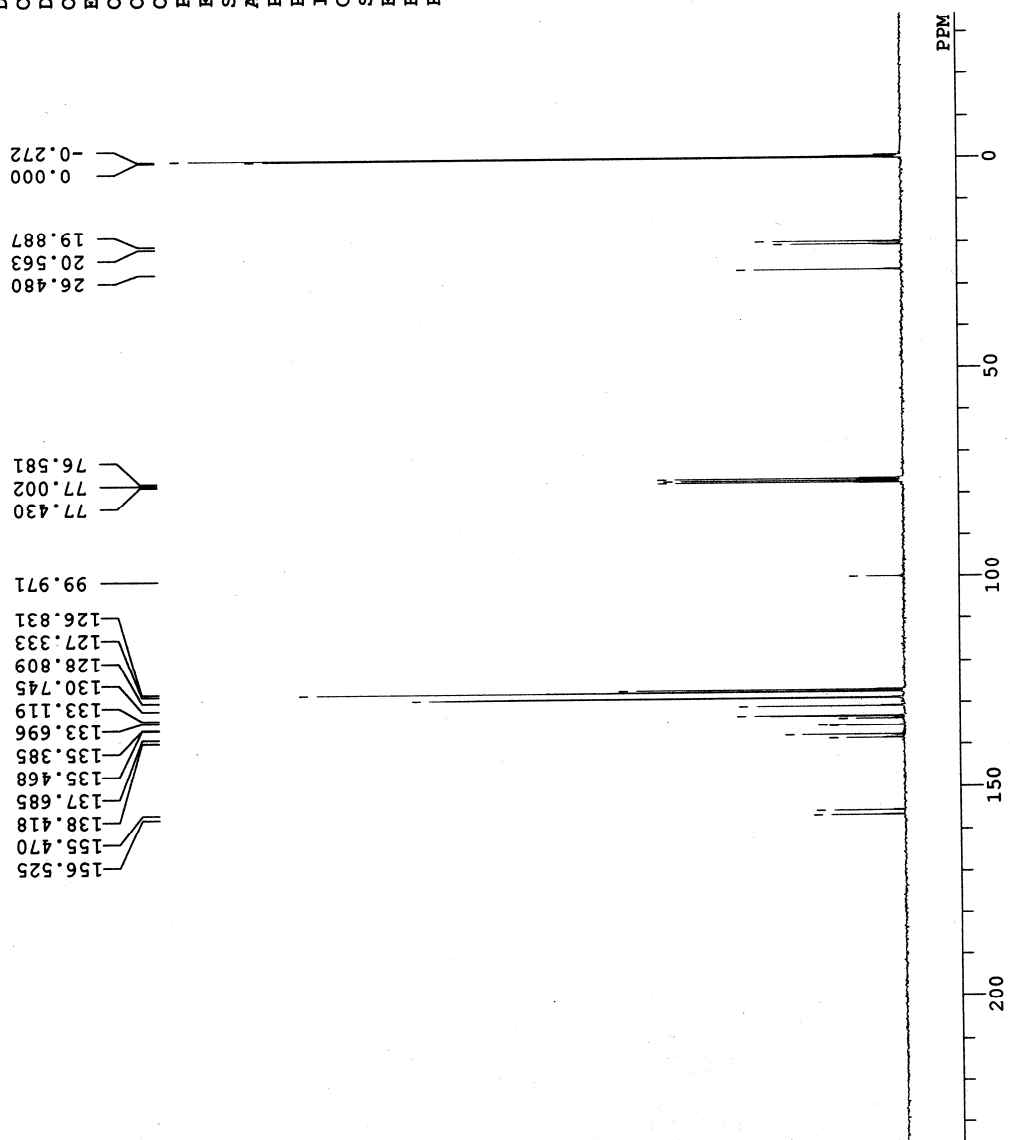
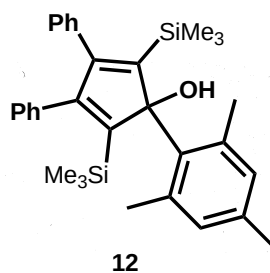


12-HNMR-noTMS



12-CNMR-withTMS

D:\LDZ\138\TMS-C.als
 COMNT Mon Jan 08 18:53:00 2007
 DATIM 13C
 OBNUC BCM
 EXMOD 75.45 MHz
 OBFRQ 124.00 KHz
 OBSET 1840.0 Hz
 OBFIN 32768
 POINT 20408.1 Hz
 FREQU 256
 SCANS 1.606 sec
 ACQTM 1.394 sec
 PD 5.0 us
 PW1 1H
 IRNUC 24.3 c
 CTEMP CDCL3
 SLYNT 0.00 ppm
 EXREF 0.62 Hz
 BF 26
 RGAIN



12-CNMR-no-TMS

D:\LDZ\138\C.als
 COMNT Mon Jan 08 12:41:47 2007
 DATIM 13C
 OBNUC BCM
 EXMOD
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 2024
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 5.0 us
 IRNUC 1H
 CTEMP 23.9 C
 SLYNT CDCL3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 26

